opening alignment is no longer within the specifications of Figure 2-2 and Figure 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings, or recalibrate the assembly.

10.2 Standard Pitot Tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 6.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

10.3 Temperature Sensors.

10.3.1 After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouplepotentiometer systems, and other sensors at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405 °C (761 °F), use an ASTM mercury-inglass reference thermometer, or equivalent, as a reference. Alternatively, either a reference thermocouple and potentiometer (calibrated against NIST standards) or thermometric fixed points (e.g., ice bath and boiling water, corrected for barometric pressure) may be used. For temperatures above 405°C (761 °F), use a reference thermocouplepotentiometer system calibrated against NIST standards or an alternative reference, subject to the approval of the Administrator.

10.3.2 The temperature data recorded in the field shall be considered valid. If, during calibration, the absolute temperature measured with the sensor being calibrated and the reference sensor agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

10.4 Barometer. Calibrate the barometer used against a mercury barometer.

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after final calculation.

12.1 Nomenclature.

A = Cross-sectional area of stack, m²

 B_{ws} = Water vapor in the gas stream (from Method 4 (reference method) or Method 5), proportion by volume.

 C_p = Pitot tube coefficient, dimensionless.

 $C_{p(s)}$ = Type S pitot tube coefficient, dimensionless.

 $C_{p(std)}$ = Standard pitot tube coefficient: use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 6.7.1 to 6.7.5 of this method.

D_e = Equivalent diameter.

 $K = 0.127 \text{ mm } H_2O \text{ (metric units)}. 0.005$ in. H_2O (English units).

 K_p = Velocity equation constant.

 M_d = Molecular weight of stack gas, dry basis (see Section 8.6), g/g-mole (lb/ lb-mole).

 M_s = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

n = Total number of traverse points.

 P_{bar} = Barometric pressure at measurement site, mm Hg (in. Hg).

 P_g = Stack static pressure, mm Hg (in.

 P_s = Absolute stack pressure ($P_{bar} + P_g$), mm Hg (in. Hg),

 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

 Q_{sd} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

T = Sensitivity factor for differential pressure gauges.

 T_s = Stack temperature, °C (°F).

 $T_{s(abs)}$ = Absolute stack temperature, °K (°R).

= $273 + T_s$ for metric units,

= $460 + T_s$ for English units.

 T_{std} = Standard absolute temperature, 293 °K (528 °R).

V_s = Average stack gas velocity, m/sec (ft/sec).

W = Width.

 $\Delta p = Velocity head of stack gas, mm$ H_2O (in. H_{20}).

 Δp_i = Individual velocity head reading at traverse point "i", mm (in.) H₂O.

 Δp_{std} = Velocity head measured by the standard pitot tube, cm (in.) H₂O.

 Δp_s = Velocity head measured by the Type S pitot tube, cm (in.) H_2O .

3600 = Conversion Factor, sec/hr.

18.0 = Molecular weight of water, g/gmole (lb/lb-mole).

12.2 Calculate T as follows:

$$T = \frac{\sum_{i=1}^{n} \sqrt{\Delta p_i + K}}{\sum_{i=1}^{n} \sqrt{\Delta p_i}}$$
 Eq. 2-1

12.3 Calculate De as follows:

$$D_{e} = \frac{2LW}{L+W} \qquad Eq. \ 2-2$$

12.4 Calibration of Type S Pitot Tube.

12.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 10.1.3, calculate the value of the Type S pitot tube coefficient according to Equation 2–3:

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p}}$$
 Eq. 2-3

12.4.2 Calculate $\overline{C}_{p(A)},$ the mean Aside coefficient, and $\overline{C}_{p(B)},$ the mean Bside coefficient. Calculate the difference between these two average values.

12.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from $\overline{C}_{p(A)}$, and the deviation of each of the three B-side values of $C_{p(s)}$ from $\overline{C}_{p(B)}$, using Equation 2–4:

Deviation =
$$C_{p(s)} - \overline{C}_{p(A \text{ or } B)}$$
 Eq. 2-4

12.4.4 Calculate σ the average deviation from the mean, for both the A and B sides of the pitot tube. Use Equation 2–5:

$$\sigma_{A \text{ or } B} = \frac{\sum_{i=1}^{3} \left| C_{p(s)} - \overline{C}_{p(A \text{ or } B)} \right|}{3}$$
 Eq. 2-5

 $M_s = M_d (1 - B_{ws}) + 18.0 B_{ws}$ Eq. 2-6 12.6 Average Stack Gas Velocity.

$$V_{s} = K_{p}C_{p}\sqrt{\Delta p_{avg}}\sqrt{\frac{T_{s(abs)}}{P_{s}M_{s}}}$$
 Eq. 2-7

$$34.97 \frac{\text{m}}{\text{sec}} \left[\frac{(\text{g/g} \cdot \text{mole})(\text{mmHg})}{(^{\circ} \text{K})(\text{mmH}_{2}\text{O})} \right]^{\frac{1}{2}} \qquad \text{Metric}$$

$$85.49 \frac{\text{m}}{\text{sec}} \left[\frac{\text{(1b/1b-mole)(in.Hg)}}{\text{(°R)(in. H2O)}} \right]^{\frac{1}{2}}$$
 English

12.7 Average Stack Gas Dry Volumetric Flow Rate.

Q = 3600(1 - B_{ws})v_sA
$$\left| \frac{T_{std}P_s}{T_{s(abs)}P_{std}} \right|$$
 Eq. 2-8

- 13.0 Method Performance [Reserved]
- 14.0 Pollution Prevention [Reserved]
- 15.0 Waste Management [Reserved]
- 16.0 References
- 1. Mark, L.S. Mechanical Engineers' Handbook. New York. McGraw-Hill Book Co., Inc. 1951.
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- 14. Vollaro, R.F. The Use of Type S Pitot Tubes for the Measurement of Low

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- 15. Smith, Marvin L. Velocity Calibration of EPA Type Source Sampling Probe. United Technologies Corporation, Pratt and Whitney Aircraft Division, East Hartford, CT. 1975.
- 16. Vollaro, R.F. Recommended Procedure for Sample Traverses in Ducts Smaller than 12 Inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, NC. November 1976.
- 17. Ower, E. and R.C. Pankhurst. The Measurement of Air Flow, 4th Ed. London, Pergamon Press. 1966.
- 18. Vollaro, R.F. A Survey of Commercially Available Instrumentation for the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, NC. November 1976. (Unpublished Paper).
- 19. Gnyp, A.W., et al. An Experimental Investigation of the Effect of Pitot Tube-Sampling Probe Configurations on the Magnitude of the S Type Pitot Tube Coefficient for Commercially Available Source Sampling Probes. Prepared by the University of Windsor for the Ministry of the Environment, Toronto, Canada. February 1975.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

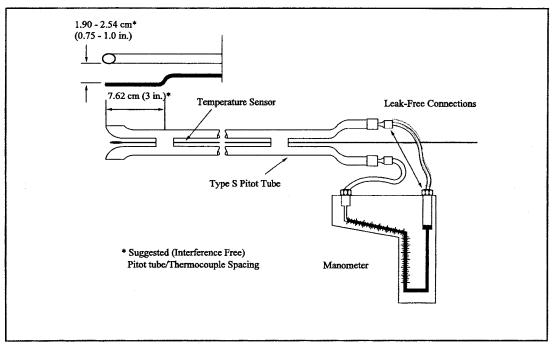


Figure 2-1. Type S Pitot Tube Manometer Assembly.

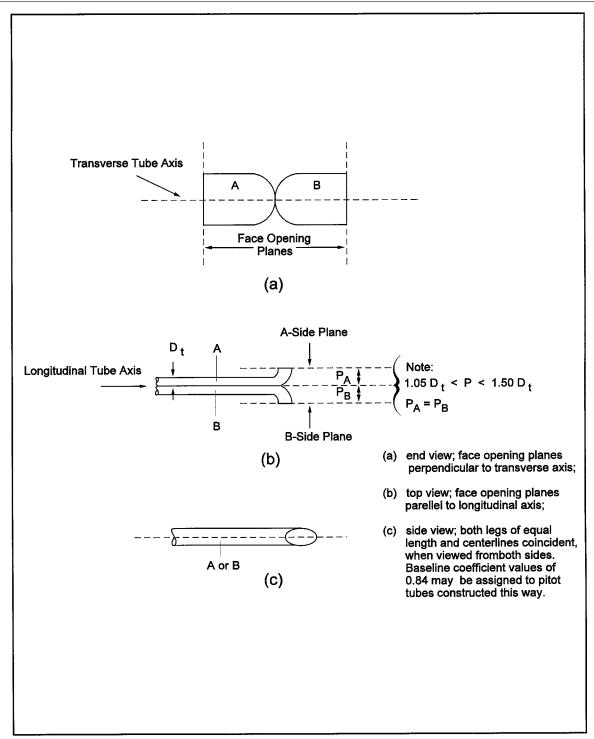


Figure 2-2. Properly Constructed Type S Pitot Tube.

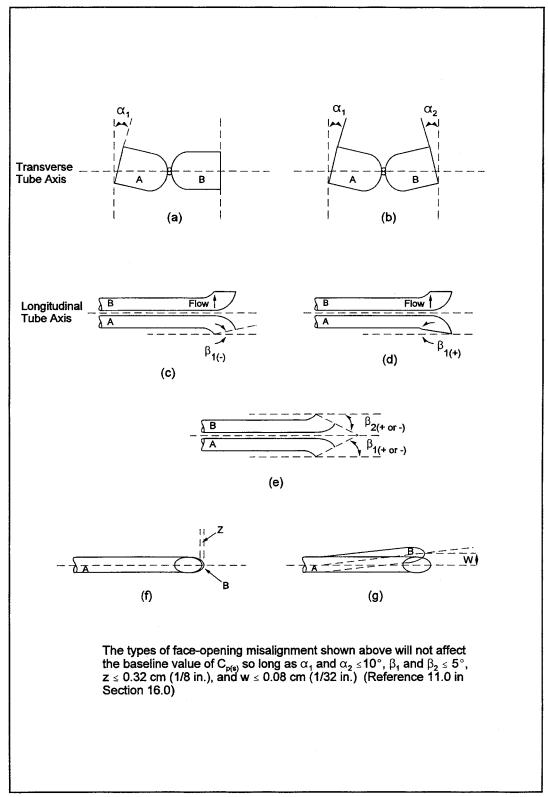


Figure 2-3. Types of face-opening misalignments that can result from field use or improper construction of type S pitot tubes.

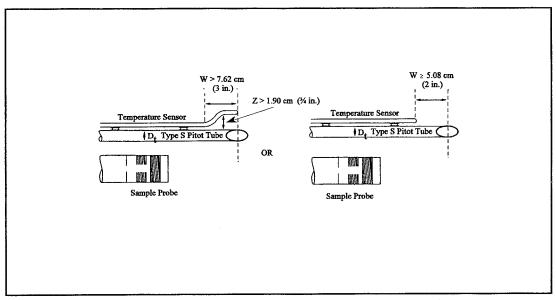


Figure 2-4. Proper temperature sensor placement to prevent interference; $D_{\rm t}$ between 0.48 and 0.95 cm (3/16 and 3/8 in).

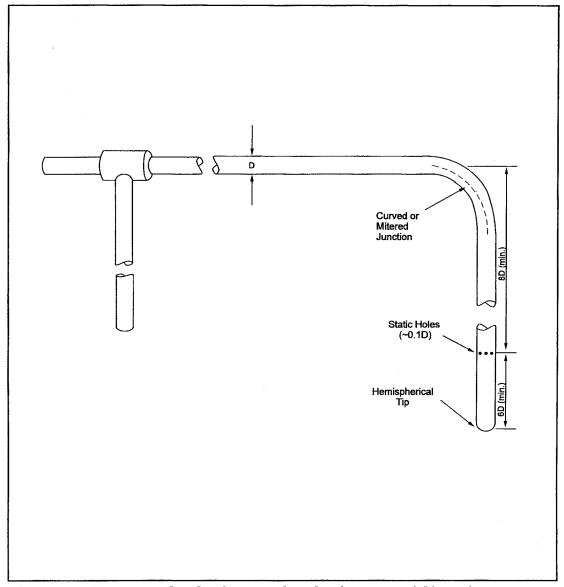


Figure 2-5. Standard pitot tube design specifications.

PLANT
DATE
RUN NO.
STACK DIA. OR DIMENSIONS, m (in.)
BAROMETRIC PRESS., mm Hg (in. Hg)
CROSS SECTIONAL AREA, m² (ft²)
OPERATORS
PITOT TUBE I.D. NO.
AVG. COEFFICIENT, Cp =
LAST DATE CALIBRATED

Traverse	Vel. Hd., Δp mm (in.) H_2O	Stack temperature		Pg	(Am)1/
Pt. No.	$\begin{array}{c} mm \ (in.) \\ H_2 O \end{array}$	T _s , °C (°F)	T _s , T _s , °C (°F) °K (°R)	Pg mm Hg (in. Hg)	(Δp)½
	Average(1)				

Figure 2–6. Velocity Traverse Data

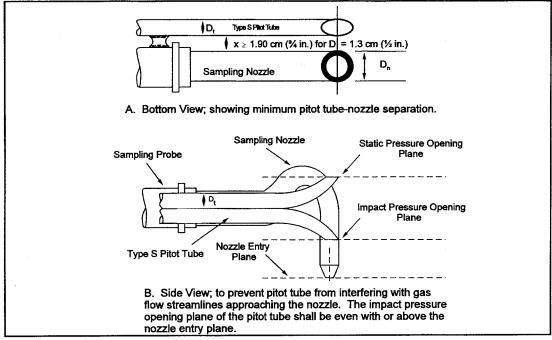


Figure 2-7. Proper pitot tube-sampling nozzle configuration.

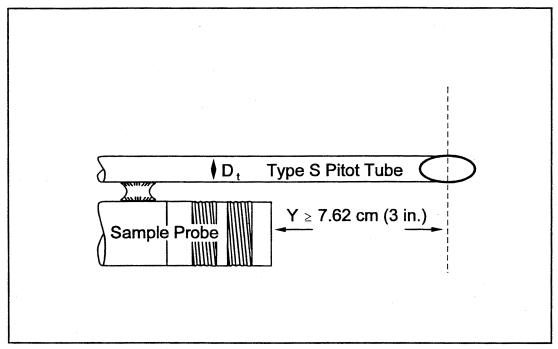


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in).

PITOT TUBE IDENTIFICATION NUMBER:

DATE:

CALIBRATED BY:

"A" SIDE CALIBRATION

Run No.	$\begin{array}{c} \Delta P_{std} \\ \text{cm H}_2\text{O} \\ \text{(in H}_2\text{O)} \end{array}$	$\begin{array}{c} \Delta P(_s) \\ \text{cm H}_2 \text{O} \\ \text{(in H}_2 \text{O)} \end{array}$	$C_{p(s)}$	$\begin{array}{c} \text{Deviation} \\ C_{p(s)} \!\!-\!\! C_p(A) \end{array}$
1				
2				
3				
		C _{p, avg} (SIDE A)		

"B" SIDE CALIBRATION

Run No.	$\begin{array}{c} \Delta P_{std} \\ \text{cm H}_2 \text{O} \\ \text{(in H}_2 \text{O)} \end{array}$	$\begin{array}{c} \Delta P_{(s)} \\ \text{cm H}_2 \text{O} \\ \text{(in H}_2 \text{O)} \end{array}$	$C_{p(s)}$	$\begin{array}{c} \text{Deviation} \\ C_{p(s)} C_p(B) \end{array}$
1				
2				
3				
		C _{p, avg} (SIDE B)		

$$\sigma_{A \text{ or } B} = \frac{\sum_{i=1}^{3} \left| C_{p(s)} - \overline{C}_{p(A \text{ or } B)} \right|}{3}$$
 Eq. 2-5

[Cp, avg (side A)—Cp, avg (side B)]*

Figure 2–9. Pitot Tube Calibration Data

^{*}Must be less than or equal to 0.01

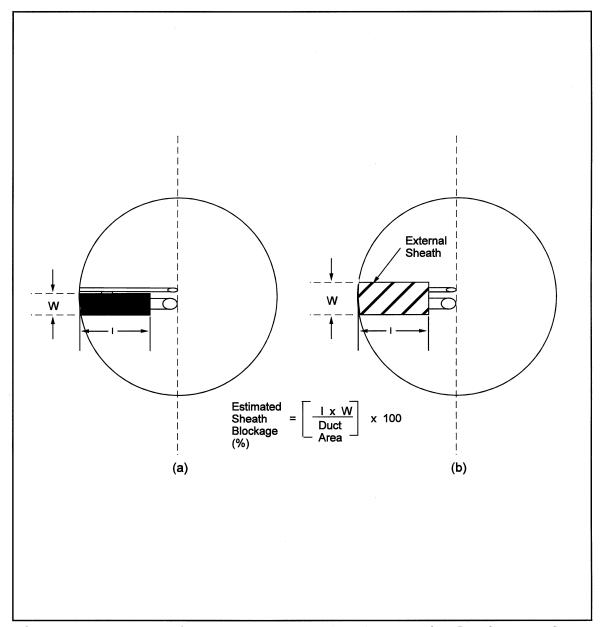


Figure 2-10. Projected-area models for typical pitot tube assemblies.

Method 2A—Direct Measurement of Gas Volume Through Pipes and Small Ducts

Note: This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2.

1.0 Scope and Application

- 1.1 This method is applicable for the determination of gas flow rates in pipes and small ducts, either in-line or at exhaust positions, within the temperature range of 0 to 50 °C (32 to 122 °F).
- 1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

- 2.1 A gas volume meter is used to measure gas volume directly.

 Temperature and pressure measurements are made to allow correction of the volume to standard conditions.
- 3.0 Definitions [Reserved]
- 4.0 Interferences [Reserved]
- 5.0 Safety
- 5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may

not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

- 6.1 Gas Volume Meter. A positive displacement meter, turbine meter, or other direct measuring device capable of measuring volume to within 2 percent. The meter shall be equipped with a temperature sensor (accurate to within ±2 percent of the minimum absolute temperature) and a pressure gauge (accurate to within ±2.5 mm Hg). The manufacturer's recommended capacity of the meter shall be sufficient for the expected maximum and minimum flow rates for the sampling conditions. Temperature, pressure, corrosive characteristics, and pipe size are factors necessary to consider in selecting a suitable gas meter.
- 6.2 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within ±2.5 mm Hg.

Note: In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

- 6.3 Stopwatch. Capable of measurement to within 1 second.
- 7.0 Reagents and Standards [Reserved]
- 8.0 Sample Collection and Analysis
- 8.1 Installation. As there are numerous types of pipes and small ducts that may be subject to volume measurement, it would be difficult to describe all possible installation schemes. In general, flange fittings should be used for all connections wherever possible. Gaskets or other seal materials should be used to assure leaktight connections. The volume meter should be located so as to avoid severe vibrations and other factors that may affect the meter calibration.
 - 8.2 Leak Test.
- 8.2.1 A volume meter installed at a location under positive pressure may be leak-checked at the meter connections by using a liquid leak detector solution containing a surfactant. Apply a small amount of the solution to the connections. If a leak exists, bubbles

- will form, and the leak must be corrected.
- 8.2.2 A volume meter installed at a location under negative pressure is very difficult to test for leaks without blocking flow at the inlet of the line and watching for meter movement. If this procedure is not possible, visually check all connections to assure leaktight seals.
 - 8.3 Volume Measurement.
- 8.3.1 For sources with continuous, steady emission flow rates, record the initial meter volume reading, meter temperature(s), meter pressure, and start the stopwatch. Throughout the test period, record the meter temperatures and pressures so that average values can be determined. At the end of the test, stop the timer, and record the elapsed time, the final volume reading, meter temperature, and pressure. Record the barometric pressure at the beginning and end of the test run. Record the data on a table similar to that shown in Figure 2A–1.
- 8.3.2 For sources with noncontinuous, non-steady emission flow rates, use the procedure in Section 8.3.1 with the addition of the following: Record all the meter parameters and the start and stop times corresponding to each process cyclical or noncontinuous event.
- 9.0 Quality Control

Section	Quality control measure	Effect
10.1–10.4	Sampling equipment calibration	Ensure accurate measurement of stack gas flow rate, sample volume.

10.0 Calibration and Standardization

10.1 Volume Meter.

- 10.1.1 The volume meter is calibrated against a standard reference meter prior to its initial use in the field. The reference meter is a spirometer or liquid displacement meter with a capacity consistent with that of the test meter.
- 10.1.2 Alternatively, a calibrated, standard pitot may be used as the reference meter in conjunction with a wind tunnel assembly. Attach the test meter to the wind tunnel so that the total flow passes through the test meter. For each calibration run, conduct a 4-point traverse along one stack diameter at a position at least eight diameters of straight tunnel downstream and two diameters upstream of any bend, inlet, or air mover. Determine the traverse point locations as specified in Method 1. Calculate the reference volume using the velocity values following the

procedure in Method 2, the wind tunnel cross-sectional area, and the run time.

- 10.1.3 Set up the test meter in a configuration similar to that used in the field installation (*i.e.*, in relation to the flow moving device). Connect the temperature sensor and pressure gauge as they are to be used in the field. Connect the reference meter at the inlet of the flow line, if appropriate for the meter, and begin gas flow through the system to condition the meters. During this conditioning operation, check the system for leaks.
- 10.1.4 The calibration shall be performed during at least three different flow rates. The calibration flow rates shall be about 0.3, 0.6, and 0.9 times the rated maximum flow rate of the test meter.
- 10.1.5 For each calibration run, the data to be collected include: reference meter initial and final volume readings, the test meter initial and final volume reading, meter average temperature and pressure, barometric pressure, and run

time. Repeat the runs at each flow rate at least three times.

- 10.1.6 Calculate the test meter calibration coefficient as indicated in Section 12.2.
- 10.1.7 Compare the three Y_m values at each of the flow rates tested and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra runs may be required to complete this requirement. If this specification cannot be met in six successive runs, the test meter is not suitable for use. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications are met at all the flow rates, average all the $Y_{\rm m}$ values from runs meeting the specifications to obtain an average meter calibration coefficient, Y_m.
- 10.1.8 The procedure above shall be performed at least once for each volume meter. Thereafter, an abbreviated calibration check shall be completed

following each field test. The calibration of the volume meter shall be checked with the meter pressure set at the average value encountered during the field test. Three calibration checks (runs) shall be performed using this average flow rate value. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of flow as described above.

Note: If the volume meter calibration coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (*i.e.*, before or after) gives the greater value of pollutant emission rate.

10.2 Temperature Sensor. After each test series, check the temperature sensor at ambient temperature. Use an American Society for Testing and

Materials (ASTM) mercury-in-glass reference thermometer, or equivalent, as a reference. If the sensor being checked agrees within 2 percent (absolute temperature) of the reference, the temperature data collected in the field shall be considered valid. Otherwise, the test data shall be considered invalid or adjustments of the results shall be made, subject to the approval of the Administrator.

10.3 Barometer. Calibrate the barometer used against a mercury barometer prior to the field test.

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation. 12.1 Nomenclature.

f = Final reading.

i = Initial reading.

 P_{bar} = Barometric pressure, mm Hg.

 $P_{\rm g}$ = Average static pressure in volume meter, mm Hg.

 Q_s = Gas flow rate, m^3 /min, standard conditions.

s = Standard conditions, 20°C and 760 mm Hg.

 T_r = Reference meter average temperature, °K (°R).

 T_m = Test meter average temperature, °K (°R).

 V_r = Reference meter volume reading,

 V_m = Test meter volume reading, m^3 .

 Y_m = Test meter calibration coefficient, dimensionless.

 θ = Elapsed test period time, min.

12.2 Test Meter Calibration Coefficient.

$$Y_{m} = \frac{\left(V_{r_{f}} - V_{r_{i}}\right) P_{b} T_{r(abs)}}{\left(V_{m_{f}} - V_{m_{i}}\right) \left(P_{b} + P_{g}\right) T_{m(abs)}}$$
 Eq. 2A-1

12.3 Volume.

$$Y_{m_s} = Y_m \left[\frac{(P_{bar} + P_g)(V_{m_f} - V_{m_i})(293 \text{ °K})}{(T_m)(760 \text{ mmHg})} \right]$$
 Eq. 2-2

12.4 Gas Flow Rate.

$$Q_s = \frac{V_{m_s}}{\theta} \qquad \text{Eq. } 2A-3$$

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

1. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. APTD–0576. March 1972.

2. Wortman, Martin, R. Vollaro, and P.R. Westlin. Dry Gas Volume Meter Calibrations. Source Evaluation Society Newsletter. Vol. 2, No. 2. May 1977.

3. Westlin, P.R., and R.T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards. Source Evaluation Society Newsletter. Vol. 3, No. 1. February 1978. 17.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

Method 2B—Determination of Exhaust Gas Volume Flow Rate From Gasoline Vapor Incinerators

Note: This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 2A, Method 10, Method 25A, Method 25B.

1.0 Scope and Application

1.1 This method is applicable for the determination of exhaust volume flow rate from incinerators that process gasoline vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). It is assumed that the amount of auxiliary fuel is negligible.

1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Organic carbon concentration and volume flow rate are measured at the incinerator inlet using either Method 25A or Method 25B and Method 2A, respectively. Organic carbon, carbon dioxide (CO₂), and carbon monoxide (CO) concentrations are measured at the outlet using either Method 25A or Method 25B and Method 10, respectively. The ratio of total carbon at the incinerator inlet and outlet is multiplied by the inlet volume to determine the exhaust volume flow rate.

3.0 Definitions

Same as Section 3.0 of Method 10 and Method 25A.

4.0 Interferences

Same as Section 4.0 of Method 10.

5.0 Safety

5.1 This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Same as Section 6.0 of Method 2A, Method 10, and Method 25A and/or Method 25B as applicable, with the addition of the following:

6.1 This analyzer must meet the specifications set forth in Section 6.1.2 of Method 10, except that the span shall be 15 percent CO₂ by volume.

7.0 Reagents and Standards

Same as Section 7.0 of Method 10 and Method 25A, with the following addition and exceptions:

- 7.1 Carbon Dioxide Analyzer Calibration. CO_2 gases meeting the specifications set forth in Section 7 of Method 6C are required.
- 7.2 Hydrocarbon Analyzer Calibration. Methane shall not be used as a calibration gas when performing this method.
- 7.3 Fuel Gas. If Method 25B is used to measure the organic carbon concentrations at both the inlet and exhaust, no fuel gas is required.

8.0 Sample Collection and Analysis

8.1 Pre-test Procedures. Perform all pre-test procedures (e.g., system performance checks, leak checks) necessary to determine gas volume flow rate and organic carbon concentration in the vapor line to the incinerator inlet and to determine organic carbon, carbon monoxide, and carbon dioxide concentrations at the incinerator exhaust, as outlined in Method 2A, Method 10, and Method 25A and/or Method 25B as applicable.

8.2 Sampling. At the beginning of the test period, record the initial parameters for the inlet volume meter according to the procedures in Method 2A and mark all of the recorder strip charts to indicate the start of the test. Conduct sampling and analysis as outlined in Method 2A, Method 10, and Method 25A and/or Method 25B as applicable. Continue recording inlet organic and exhaust CO2, CO, and organic concentrations throughout the test. During periods of process interruption and halting of gas flow, stop the timer and mark the recorder strip charts so that data from this interruption are not included in the calculations. At the end of the test period, record the final parameters for the inlet volume meter and mark the end on all of the recorder strip charts.

8.3 Post-test Procedures. Perform all post-test procedures (e.g., drift tests, leak checks), as outlined in Method 2A, Method 10, and Method 25A and/or Method 25B as applicable.

9.0 Quality Control

Same as Section 9.0 of Method 2A, Method 10, and Method 25A.

10.0 Calibration and Standardization

Same as Section 10.0 of Method 2A, Method 10, and Method 25A.

Note: If a manifold system is used for the exhaust analyzers, all the analyzers and sample pumps must be operating when the analyzer calibrations are performed.

10.1 If an analyzer output does not meet the specifications of the method, invalidate the test data for the period. Alternatively, calculate the exhaust volume results using initial calibration data and using final calibration data and report both resulting volumes. Then, for emissions calculations, use the volume measurement resulting in the greatest emission rate or concentration.

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

Carry out the calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation.

12.1 Nomenclature.

- Co_e = Mean carbon monoxide concentration in system exhaust, ppm.
- $(CO_2)_2$ = Ambient carbon dioxide concentration, ppm (if not measured during the test period, may be assumed to equal 300 ppm).
- (CO₂)_e = Mean carbon dioxide concentration in system exhaust, ppm.
- HC_e = Mean organic concentration in system exhaust as defined by the calibration gas, ppm.
- Hc_i = Mean organic concentration in system inlet as defined by the calibration gas, ppm.
- K_e = Hydrocarbon calibration gas factor for the exhaust hydrocarbon analyzer, unitless [equal to the number of carbon atoms per molecule of the gas used to calibrate the analyzer (2 for ethane, 3 for propane, etc.)].
- K_i = Hydrocarbon calibration gas factor for the inlet hydrocarbon analyzer, unitless.
- V_{es} = Exhaust gas volume, m^3 .
- V_{is} = Inlet gas volume, m^3 .
- Q_{es} = Exhaust gas volume flow rate, m^3/min .
- Q_{is} = Inlet gas volume flow rate, m³/ min.
- θ = Sample run time, min.
- s = Standard conditions: 20 °C, 760 mm Hg.
- 12.2 Concentrations. Determine mean concentrations of inlet organics, outlet CO₂, outlet CO, and outlet organics according to the procedures in the respective methods and the analyzers' calibration curves, and for the time intervals specified in the applicable regulations.
- 12.3 Exhaust Gas Volume. Calculate the exhaust gas volume as follows:

$$V_{es} = V_{is} \frac{K_i(HC_i)}{K_e(HC_e) + [(CO_2)_e - (CO_2)_a] + CO_e}$$
 Eq. 2B-1

12.4 Exhaust Gas Volume Flow Rate. Calculate the exhaust gas volume flow rate as follows:

$$Q_{es} = \frac{V_{es}}{\Theta}$$
 Eq. 2B-2

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Section 16.0 of Method 2A, Method 10, and Method 25A.

17.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

Method 2C—Determination of Gas Velocity and Volumetric Flow Rate in Small Stacks or Ducts (Standard Pitot Tube)

Note: This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Method 1, Method 2.

1.0 Scope and Application

1.1 This method is applicable for the determination of average velocity and volumetric flow rate of gas streams in small stacks or ducts. Limits on the applicability of this method are identical to those set forth in Method 2, Section 1.0, except that this method is limited to stationary source stacks or ducts less than about 0.30 meter (12 in.) in diameter, or 0.071 m² (113 in.²) in

cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter, or 0.0081 m^2 (12.57 in.^2) in cross-sectional area.

1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

- 2.1 The average gas velocity in a stack or duct is determined from the gas density and from measurement of velocity heads with a standard pitot tube.
- 3.0 Definitions [Reserved]
- 4.0 Interferences [Reserved]
- 5.0 Safety
- 5.1 This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.
- 6.0 Equipment and Supplies

Same as Method 2, Section 6.0, with the exception of the following:

6.1 Standard Pitot Tube (instead of Type S). A standard pitot tube which meets the specifications of Section 6.7 of Method 2. Use a coefficient of 0.99 unless it is calibrated against another standard pitot tube with a NIST-traceable coefficient (see Section 10.2 of Method 2).

6.2 Alternative Pitot Tube. A modified hemispherical-nosed pitot tube (see Figure 2C–1), which features a shortened stem and enlarged impact and static pressure holes. Use a coefficient of 0.99 unless it is calibrated as mentioned in Section 6.1 above. This pitot tube is useful in particulate liquid dropletladen gas streams when a "back purge" is ineffective.

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection and Analysis

8.1 Follow the general procedures in Section 8.0 of Method 2, except conduct the measurements at the traverse points specified in Method 1A. The static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, adequate proof that the openings of the pitot tube have not plugged during the traverse period must be furnished; this can be done by taking the velocity head (Δp) heading at the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "backpurging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (within ±5 percent) the traverse is acceptable. Otherwise, reject the run. Note that if the Δp at the final traverse point is unsuitably low, another point may be selected. If "back purging" at regular intervals is part of the procedure, then take comparative Δp readings, as above, for the last two back purges at which suitably high Δp readings are observed.

9.0 Quality Control

Section	Quality control measure	Effect
10.0	Sampling equipment calibration	Ensure accurate measurement of stack gas velocity head.

10.0 Calibration and Standardization

Same as Method 2, Sections 10.2 through 10.4.

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Calculations and Data Analysis

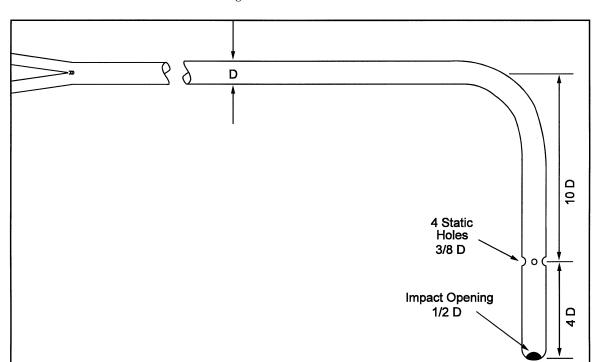
Same as Method 2, Section 12.0.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References



17.0 Tables, Diagrams, Flowcharts, and Validation Data

Figure 2C-1. Modified Hemispherical-Nosed Pitot Tube.

Method 2D—Measurement of Gas Volume Flow Rates in Small Pipes and Ducts

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, and Method 2A.

1.0 Scope and Application

- 1.1 This method is applicable for the determination of the volumetric flow rates of gas streams in small pipes and ducts. It can be applied to intermittent or variable gas flows only with particular caution.
- 1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 All the gas flow in the pipe or duct is directed through a rotameter, orifice plate or similar device to measure flow rate or pressure drop. The device has been previously calibrated in a manner that insures its proper calibration for the gas being measured. Absolute temperature and pressure measurements are made to allow

correction of volumetric flow rates to standard conditions.

- 3.0 Definitions. [Reserved]
- 4.0 Interferences. [Reserved]
- 5.0 Safety
- 5.1 This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

6.1 Gas Metering Rate or Flow Element Device. A rotameter, orifice plate, or other volume rate or pressure drop measuring device capable of measuring the stack flow rate to within ±5 percent. The metering device shall be equipped with a temperature gauge accurate to within ±2 percent of the minimum absolute stack temperature and a pressure gauge (accurate to within ±5 mm Hg). The capacity of the metering device shall be sufficient for

the expected maximum and minimum flow rates at the stack gas conditions. The magnitude and variability of stack gas flow rate, molecular weight, temperature, pressure, dewpoint, and corrosive characteristics, and pipe or duct size are factors to consider in choosing a suitable metering device.

- 6.2 Barometer. Same as Method 2, Section 6.5.
- 6.3 Stopwatch. Capable of measurement to within 1 second.
- 7.0 Reagents and Standards. [Reserved]
- 8.0 Sample Collection and Analysis
- 8.1 Installation and Leak Check. Same as Method 2A, Sections 8.1 and 8.2, respectively.
 - 8.2 Volume Rate Measurement.
- 8.2.1 Continuous, Steady Flow. At least once an hour, record the metering device flow rate or pressure drop reading, and the metering device temperature and pressure. Make a minimum of 12 equally spaced readings of each parameter during the test period. Record the barometric pressure at the beginning and end of the test period. Record the data on a table similar to that shown in Figure 2D–1.
- 8.2.2 Noncontinuous and Nonsteady Flow. Use volume rate devices with particular caution. Calibration will be affected by variation in stack gas temperature, pressure and molecular

weight. Use the procedure in Section 8.2.1 with the addition of the following: Record all the metering device

parameters on a time interval frequency sufficient to adequately profile each process cyclical or noncontinuous event. A multichannel continuous recorder may be used.

9.0 Quality Control

Section	Quality control measure	Effect
10.0	Sampling equipment calibration	Ensure accurate measurement of stack gas flow rate or sample volume.

10.0 Calibration and Standardization

Same as Method 2A, Section 10.0, with the following exception:

10.1 Gas Metering Device. Same as Method 2A, Section 10.1, except calibrate the metering device with the principle stack gas to be measured (examples: air, nitrogen) against a standard reference meter. A calibrated dry gas meter is an acceptable reference meter. Ideally, calibrate the metering device in the field with the actual gas to be metered. For metering devices that have a volume rate readout, calculate the test metering device calibration coefficient, Y_m, for each run shown in Equation 2D–2 Section 12.3.

 $\hat{1}0.2$ For metering devices that do not have a volume rate readout, refer to the manufacturer's instructions to calculate the V_{m2} corresponding to each V

10.3 Temperature Gauge. Use the procedure and specifications in Method 2A, Section 10.2. Perform the calibration at a temperature that approximates field test conditions.

10.4 Barometer. Calibrate the barometer to be used in the field test with a mercury barometer prior to the field test.

11.0 Analytical Procedure.

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

12.1 Nomenclature.

 P_{bar} = Barometric pressure, mm Hg (in. Hg).

 $P_m = \overline{T}$ est meter average static pressure, mm Hg (in. Hg).

 Q_r = Reference meter volume flow rate reading, m³/min (ft³/min).

Q_m = Test meter volume flow rate reading, m³/min (ft³/min).

 T_r = Absolute reference meter average temperature, °K (°R).

T_m = Absolute test meter average temperature, °K (°R).

 $K_1 = 0.3855$ °K/mm Hg for metric units, = 17.65 °R/in. Hg for English units. 12.2 Gas Flow Rate. $Q_{s} = K_{1}Y_{m}Q_{m} \frac{(P_{bar} + P_{m})}{T_{m}}$ Eq. 2D-1

12.3 Test Meter Device Calibration Coefficient. Calculation for testing metering device calibration coefficient, Y_m .

$$Y_{m} = \frac{Q_{r}T_{r}P_{bar}}{Q_{m}T_{m}(P_{bar} + P_{m})}$$
 Eq. 2D-2

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

1. Spink, L.K. Principles and Practice of Flowmeter Engineering. The Foxboro Company. Foxboro, MA. 1967.

2. Benedict, R.P. Fundamentals of Temperature, Pressure, and Flow Measurements. John Wiley & Sons, Inc. New York, NY. 1969.

3. Orifice Metering of Natural Gas. American Gas Association. Arlington, VA. Report No. 3. March 1978. 88 pp.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

Plant
Date
Run No.
Sample location
Barometric pressure (mm Hg):
Start
Finish
Operators
Metering device No.
Calibration coefficient
Calibration gas
Date to recalibrate

Time	Flow rate	Static Pressure	Temp	erature
rime	Flow rate reading	Static Pressure [mm Hg (in. Hg)]	°C (°F)	°K (°R)
Average				

Figure 2D-1. Volume Flow Rate Measurement Data

Method 2E—Determination of Landfill Gas Production Flow Rate

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Methods 2 and 3C.

1.0 Scope and Application

- 1.1 Applicability. This method applies to the measurement of landfill gas (LFG) production flow rate from municipal solid waste landfills and is used to calculate the flow rate of nonmethane organic compounds (NMOC) from landfills.
- 1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

- 2.1 Extraction wells are installed either in a cluster of three or at five dispersed locations in the landfill. A blower is used to extract LFG from the landfill. LFG composition, landfill pressures, and orifice pressure differentials from the wells are measured and the landfill gas production flow rate is calculated.
- 3.0 Definitions [Reserved]
- 4.0 Interferences [Reserved]
- 5.0 Safety
- 5.1 Since this method is complex, only experienced personnel should perform the test. Landfill gas contains methane, therefore explosive mixtures may exist at or near the landfill. It is advisable to take appropriate safety precautions when testing landfills, such as refraining from smoking and installing explosion-proof equipment.

6.0 Equipment and Supplies

- 6.1 Well Drilling Rig. Capable of boring a 0.61 m (24 in.) diameter hole into the landfill to a minimum of 75 percent of the landfill depth. The depth of the well shall not extend to the bottom of the landfill or the liquid level.
- 6.2 Gravel. No fines. Gravel diameter should be appreciably larger than perforations stated in Sections 6.10 and 8.2.
 - 6.3 Bentonite.
- 6.4 Backfill Material. Clay, soil, and sandy loam have been found to be acceptable.

- 6.5 Extraction Well Pipe. Minimum diameter of 3 in., constructed of polyvinyl chloride (PVC), high density polyethylene (HDPE), fiberglass, stainless steel, or other suitable nonporous material capable of transporting landfill gas.
- 6.6 Above Ground Well Assembly. Valve capable of adjusting gas flow, such as a gate, ball, or butterfly valve; sampling ports at the well head and outlet; and a flow measuring device, such as an in-line orifice meter or pitot tube. A schematic of the aboveground well head assembly is shown in Figure 2E–1.
- 6.7 Cap. Constructed of PVC or HDPE.
- 6.8 Header Piping. Constructed of PVC or HDPE.
- 6.9 Auger. Capable of boring a 0.15-to 0.23-m (6-to 9-in.) diameter hole to a depth equal to the top of the perforated section of the extraction well, for pressure probe installation.
- 6.10 Pressure Probe. Constructed of PVC or stainless steel (316), 0.025-m (1-in.). Schedule 40 pipe. Perforate the bottom two-thirds. A minimum requirement for perforations is slots or holes with an open area equivalent to four 0.006-m (1/4-in.) diameter holes spaced 90° apart every 0.15 m (6 in.).
- 6.11 Blower and Flare Assembly. Explosion-proof blower, capable of extracting LFG at a flow rate of 8.5 m ³/min (300 ft ³/min), a water knockout, and flare or incinerator.
- 6.12 Standard Pitot Tube and Differential Pressure Gauge for Flow Rate Calibration with Standard Pitot. Same as Method 2, Sections 6.7 and 6.8.
- 6.13 Orifice Meter. Orifice plate, pressure tabs, and pressure measuring device to measure the LFG flow rate.
- 6.14 Barometer. Same as Method 4, Section 6.1.5.
- 6.15 Differential Pressure Gauge. Water-filled U-tube manometer or equivalent, capable of measuring within 0.02 mm Hg (0.01 in. H_2O), for measuring the pressure of the pressure probes.
- 7.0 Reagents and Standards. Not Applicable
- 8.0 Sample Collection, Preservation, Storage, and Transport
- 8.1 Placement of Extraction Wells. The landfill owner or operator may install a single cluster of three extraction wells in a test area or space five equal-volume wells over the landfill. The cluster wells are recommended but may be used only if the composition, age of the refuse, and

- the landfill depth of the test area can be determined.
- 8.1.1 Cluster Wells. Consult landfill site records for the age of the refuse, depth, and composition of various sections of the landfill. Select an area near the perimeter of the landfill with a depth equal to or greater than the average depth of the landfill and with the average age of the refuse between 2 and 10 years old. Avoid areas known to contain nondecomposable materials, such as concrete and asbestos. Locate the cluster wells as shown in Figure 2E–2.
- 8.1.1.1 The age of the refuse in a test area will not be uniform, so calculate a weighted average age of the refuse as shown in Section 12.2.
- 8.1.2 Equal Volume Wells. Divide the sections of the landfill that are at least 2 years old into five areas representing equal volumes. Locate an extraction well near the center of each area.
- 8.2 Installation of Extraction Wells. Use a well drilling rig to dig a 0.6 m (24 in.) diameter hole in the landfill to a minimum of 75 percent of the landfill depth, not to extend to the bottom of the landfill or the liquid level. Perforate the bottom two thirds of the extraction well pipe. A minimum requirement for perforations is holes or slots with an open area equivalent to 0.01-m (0.5-in.) diameter holes spaced 90° apart every 0.1 to 0.2 m (4 to 8 in.). Place the extraction well in the center of the hole and backfill with gravel to a level 0.30 m (1 ft) above the perforated section. Add a layer of backfill material 1.2 m (4 ft) thick. Add a layer of bentonite 0.9 m (3 ft) thick, and backfill the remainder of the hole with cover material or material equal in permeability to the existing cover material. The specifications for extraction well installation are shown in Figure 2E-3.
- 8.3 Pressure Probes. Shallow pressure probes are used in the check for infiltration of air into the landfill, and deep pressure probes are use to determine the radius of influence. Locate pressure probes along three radial arms approximately 120° apart at distances of 3, 15, 30, and 45 m (10, 50, 100, and 150 ft) from the extraction well. The tester has the option of locating additional pressure probes at distances every 15 m (50 feet) beyond 45 m (150 ft). Example placements of probes are shown in Figure 2E–4. The 15-, 30-, and 45-m, (50-, 100-, and 150ft) probes from each well, and any additional probes located along the three radial arms (deep probes), shall

extend to a depth equal to the top of the perforated section of the extraction wells. All other probes (shallow probes) shall extend to a depth equal to half the

depth of the deep probes.

8.3.1 Use an auger to dig a hole, 0.15- to 0.23-m (6-to 9-in.) in diameter, for each pressure probe. Perforate the bottom two thirds of the pressure probe. A minimum requirement for perforations is holes or slots with an open area equivalent to four 0.006-m (0.25-in.) diameter holes spaced 90° apart every 0.15 m (6 in.). Place the pressure probe in the center of the hole and backfill with gravel to a level 0.30 m (1 ft) above the perforated section. Add a layer of backfill material at least 1.2 m (4 ft) thick. Add a layer of bentonite at least 0.3 m (1 ft) thick, and backfill the remainder of the hole with cover material or material equal in permeability to the existing cover material. The specifications for pressure probe installation are shown in Figure 2E-5.

8.4 LFG Flow Rate Measurement. Place the flow measurement device, such as an orifice meter, as shown in Figure 2E–1. Attach the wells to the blower and flare assembly. The individual wells may be ducted to a common header so that a single blower, flare assembly, and flow meter may be used. Use the procedures in Section 10.1 to calibrate the flow meter.

8.5 Leak-Check. A leak-check of the above ground system is required for accurate flow rate measurements and for safety. Sample LFG at the well head sample port and at the outlet sample port. Use Method 3C to determine nitrogen (N₂) concentrations. Determine the difference between the well head and outlet N₂ concentrations using the formula in Section 12.3. The system passes the leak-check if the difference is

less than 10,000 ppmv.

8.6 Static Testing. Close the control valves on the well heads during static testing. Measure the gauge pressure (Pg) at each deep pressure probe and the barometric pressure (Pbar) every 8 hours (hr) for 3 days. Convert the gauge pressure of each deep pressure probe to absolute pressure using the equation in Section 12.4. Record as Pi (initial absolute pressure).

8.6.1 For each probe, average all of the 8-hr deep pressure probe readings (P_i) and record as P_{ia} (average absolute pressure). P_{ia} is used in Section 8.7.5 to determine the maximum radius of influence.

8.6.2 Measure the static flow rate of each well once during static testing.

- 8.7 Short-Term Testing. The purpose of short-term testing is to determine the maximum vacuum that can be applied to the wells without infiltration of ambient air into the landfill. The shortterm testing is performed on one well at a time. Burn all LFG with a flare or incinerator.
- 8.7.1 Use the blower to extract LFG from a single well at a rate at least twice the static flow rate of the respective well measured in Section 8.6.2. If using a single blower and flare assembly and a common header system, close the control valve on the wells not being measured. Allow 24 hr for the system to stabilize at this flow rate.
- 8.7.2 Test for infiltration of air into the landfill by measuring the gauge pressures of the shallow pressure probes and using Method 3C to determine the LFG N₂ concentration. If the LFG N₂ concentration is less than 5 percent and all of the shallow probes have a positive gauge pressure, increase the blower vacuum by 3.7 mm Hg (2 in. H₂O), wait 24 hr, and repeat the tests for infiltration. Continue the above steps of increasing blower vacuum by 3.7 mm Hg (2 in. H_2O), waiting 24 hr, and testing for infiltration until the concentration of N₂ exceeds 5 percent or any of the shallow probes have a negative gauge pressure. When this occurs, reduce the blower vacuum to the maximum setting at which the N₂ concentration was less than 5 percent and the gauge pressures of the shallow probes are positive.
- 8.7.3 At this blower vacuum, measure atmospheric pressure (Pbar) every 8 hr for 24 hr, and record the LFG flow rate (Q_s) and the probe gauge pressures (P_f) for all of the probes. Convert the gauge pressures of the deep probes to absolute pressures for each 8hr reading at Q_s as shown in Section 12.4.
- 8.7.4 For each probe, average the 8hr deep pressure probe absolute pressure readings and record as Pfa (the final average absolute pressure).
- 8.7.5 For each probe, compare the initial average pressure (Pia) from Section 8.6.1 to the final average pressure (Pfa). Determine the furthermost point from the well head along each radial arm where $P_{fa} \le P_{ia}$. This distance is the maximum radius of influence (R_m), which is the distance from the well affected by the vacuum. Average these values to determine the average maximum radius of influence
- 8.7.6 Calculate the depth (D_{st}) affected by the extraction well during

- the short term test as shown in Section 12.6. If the computed value of D_{st} exceeds the depth of the landfill, set D_{st} equal to the landfill depth.
- 8.7.7 Calculate the void volume (V) for the extraction well as shown in Section 12.7.
- 8.7.8 Repeat the procedures in Section 8.7 for each well.
- 8.8 Calculate the total void volume of the test wells (V_v) by summing the void volumes (V) of each well.
- 8.9 Long-Term Testing. The purpose of long-term testing is to extract two void volumes of LFG from the extraction wells. Use the blower to extract LFG from the wells. If a single Blower and flare assembly and common header system are used, open all control valves and set the blower vacuum equal to the highest stabilized blower vacuum demonstrated by any individual well in Section 8.7. Every 8 hr, sample the LFG from the well head sample port, measure the gauge pressures of the shallow pressure probes, the blower vacuum, the LFG flow rate, and use the criteria for infiltration in Section 8.7.2 and Method 3C to test for infiltration. If infiltration is detected, do not reduce the blower vacuum, instead reduce the LFG flow rate from the well by adjusting the control valve on the well head. Adjust each affected well individually. Continue until the equivalent of two total void volumes (V_v) have been extracted, or until $V_t = 2V_v$.
- 8.9.1 Calculate V_t, the total volume of LFG extracted from the wells, as shown in Section 12.8.
- 8.9.2 Record the final stabilized flow rate as Qf and the gauge pressure for each deep probe. If, during the long term testing, the flow rate does not stabilize, calculate Q_f by averaging the last 10 recorded flow rates.
- 8.9.3 For each deep probe, convert each gauge pressure to absolute pressure as in Section 12.4. Average these values and record as Psa. For each probe, compare P_{ia} to P_{sa}. Determine the furthermost point from the well head along each radial arm where $P_{sa} \leq P_{ia}$. This distance is the stabilized radius of influence. Average these values to determine the average stabilized radius of influence (R_{sa}) .
- 8.10 Determine the NMOC mass emission rate using the procedures in Section 12.9 through 12.15.
- 9.0 Quality Control
- 9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
10.1	LFG flow rate meter calibration	Ensures accurate measurement of LFG flow rate and sample volume

10.0 Calibration and Standardization

10.1 LFG Flow Rate Meter (Orifice) Calibration Procedure. Locate a standard pitot tube in line with an orifice meter. Use the procedures in Section 8, 12.5, 12.6, and 12.7 of Method 2 to determine the average dry gas volumetric flow rate for at least five flow rates that bracket the expected LFG flow rates, except in Section 8.1, use a standard pitot tube rather than a Type S pitot tube. Method 3C may be used to determine the dry molecular weight. It may be necessary to calibrate more than one orifice meter in order to bracket the LFG flow rates. Construct a calibration curve by plotting the pressure drops across the orifice meter for each flow rate versus the average dry gas volumetric flow rate in m³/min of the gas.

11.0 Procedures [Reserved]

12.0 Data Analysis and Calculations

12.1 Nomenclature.

A = Age of landfill, yr.

A_{avg} = Average age of the refuse tested,

 $A_i = Age$ of refuse in the ith fraction, yr. $A_r = Acceptance rate, Mg/yr.$

 C_{NMOC} = NMOC concentration, ppmv as hexane ($C_{NMOC} = C_t/6$).

 C_o = Concentration of N_2 at the outlet,

 $C_t = NMOC$ concentration, ppmv (carbon equivalent) from Method 25C.

 C_w = Concentration of N_2 at the wellhead, ppmv.

D = Depth affected by the test wells, m.

 D_{st} = Depth affected by the test wells in the short-term test, m.

e = Base number for natural logarithms (2.718).

f = Fraction of decomposable refuse in the landfill.

 f_i = Fraction of the refuse in the ith section.

k = Landfill gas generation constant, yr^{-1} .

L_o = Methane generation potential, m³/

 $L_{o}' = Revised methane generation$ potential to account for the amount of nondecomposable material in the landfill, m3/Mg.

 $M_i = Mass of refuse in the ith section,$ Mg.

 $M_r = Mass$ of decomposable refuse affected by the test well, Mg.

P_{bar} = Atmospheric pressure, mm Hg. P_f = Final absolute pressure of the deep pressure probes during short-term testing, mm Hg.

 P_{fa} = Average final absolute pressure of the deep pressure probes during short-term testing, mm Hg.

 P_{gf} = final gauge pressure of the deep pressure probes, mm Hg.

 P_{gi} = Initial gauge pressure of the deep pressure probes, mm Hg.

 P_i = Initial absolute pressure of the deep pressure probes during static testing, mm Hg.

 P_{ia} = Average initial absolute pressure of the deep pressure probes during static testing, mm Hg.

 P_s = Final absolute pressure of the deep pressure probes during long-term testing, mm Hg.

 P_{sa} = Average final absolute pressure of the deep pressure probes during long-term testing, mm Hg.

 Q_f = Final stabilized flow rate, m³/min. Q_i = LFG flow rate measured at orifice meter during the ith interval, m³/

 Q_s = Maximum LFG flow rate at each well determined by short-term test, m³/min.

 $Q_t = NMOC$ mass emission rate, m^3/min .

 $R_{\rm m}$ = Maximum radius of influence, m.

 $R_{\rm ma}$ = Average maximum radius of influence, m.

 R_s = Stabilized radius of influence for an individual well, m.

 R_{sa} = Average stabilized radius of influence, m.

 t_i = Age of section i, yr.

 $t_t = \text{Total time of long-term testing, yr.}$ t_{vi} = Time of the ith interval (usually 8),

hr.

 $V = Void volume of test well, m^3$.

 V_r = Volume of refuse affected by the test well, m³.

 V_t = Total volume of refuse affected by the long-term testing, m³.

 V_v = Total void volume affected by test wells, m³.

WD = Well depth, m.

 ρ = Refuse density, Mg/m³ (Assume 0.64 Mg/m³ if data are unavailable).

12.2 Use the following equation to calculate a weighted average age of landfill refuse.

$$A_{\text{avg}} = \sum_{i=1}^{n} f_i A_i \qquad \text{Eq. 2E-1}$$

12.3 Use the following equation to determine the difference in N₂ concentrations (ppmv) at the well head and outlet location.

Difference = $C_o - C_w$ Eq. 2E-2

12.4 Use the following equation to convert the gauge pressure (Pg) of each initial deep pressure probe to absolute pressure (P_i).

$$P_i = P_{bar} + P_{gi}$$
 Eq. 2E-3

12.5 Use the following equation to convert the gauge pressures of the deep probes to absolute pressures for each 8hr reading at Q_s.

$$P_f = P_{bar} + P_{gf}$$
 Eq. 2E-4

12.6 Use the following equation to calculate the depth (Dst) affected by the extraction well during the short-term

$$D_{st} = WD + R_{ma}$$
 Eq. 2E-5

12.7 Use the following equation to calculate the void volume for the extraction well (V).

$$V = 0.40 \,\Pi \,R_{ma}^{2} D_{st}$$
 Eq. 2E-6

12.8 Use the following equation to calculate V_t, the total volume of LFG extracted from the wells.

$$V_t = \sum_{i=1}^{n} 60 Q_i t_{vi}$$
 Eq. 2E-7

12.9 Use the following equation to calculate the depth affected by the test well. If using cluster wells, use the average depth of the wells for WD. If the value of D is greater than the depth of the landfill, set D equal to the landfill depth.

$$D = WD + R_{sa}$$
 Eq. 2E-8

12.10 Use the following equation to calculate the volume of refuse affected by the test well.

$$V_r = R_{sa}^2 \prod D$$
 Eq. 2E-9

12.11 Use the following equation to calculate the mass affected by the test well.

$$M_r = V_r \rho$$
 Eq. 2E-10

12.12 Modify Lo to account for the nondecomposable refuse in the landfill.

$$L_o' = f L_o$$
 Eq. 2E-11

12.13 In the following equation, solve for k (landfill gas generation constant) by iteration. A suggested procedure is to select a value for k, calculate the left side of the equation, and if not equal to zero, select another value for k. Continue this process until the left hand side of the equation equals zero, ± 0.001 .

$$k_e^{-k} A_{avg} - \frac{Q_f}{2 L_o M_r} = 0$$
 Eq. 2E-12

12.14 Use the following equation to determine landfill NMOC mass emission rate if the yearly acceptance

rate of refuse has been consistent (10 percent) over the life of the landfill.

$$Q_t = 2L'_oA_r(1-e^{-kA})C_{NMOC}(3.595\times10^{-9})$$
 Eq. 2E-13

12.15 Use the following equation to determine landfill NMOC mass emission rate if the acceptance rate has not been consistent over the life of the landfill.

$$Q_t = 2 k L_o' C_{NMOC} (3.595 \times 10^{-9}) \sum_{i=1}^n M_i e^{-kt_i}$$
 Eq. 2E-14

- 13.0 Method Performance. [Reserved]
- 14.0 Pollution Prevention. [Reserved]
- 15.0 Waste Management. [Reserved]
- 16.0 References
- 1. Same as Method 2, Appendix A, 40 CFR Part 60.
- 2. Emcon Associates, Methane Generation and Recovery from Landfills. Ann Arbor Science, 1982.
- 3. The Johns Hopkins University, Brown Station Road Landfill Gas Resource Assessment, Volume 1: Field Testing and Gas Recovery Projections. Laurel, Maryland: October 1982.
- 4. Mandeville and Associates, Procedure Manual for Landfill Gases Emission Testing.
- 5. Letter and attachments from Briggum, S., Waste Management of North America, to Thorneloe, S., EPA. Response to July 28,

1988 request for additional information. August 18, 1988.

6. Letter and attachments from Briggum, S., Waste Management of North America, to Wyatt, S., EPA. Response to December 7, 1988 request for additional information. January 16, 1989.

BILLING CODE 6560-50-C

17.0 Tables, Diagrams, Flowcharts, and Validation Data

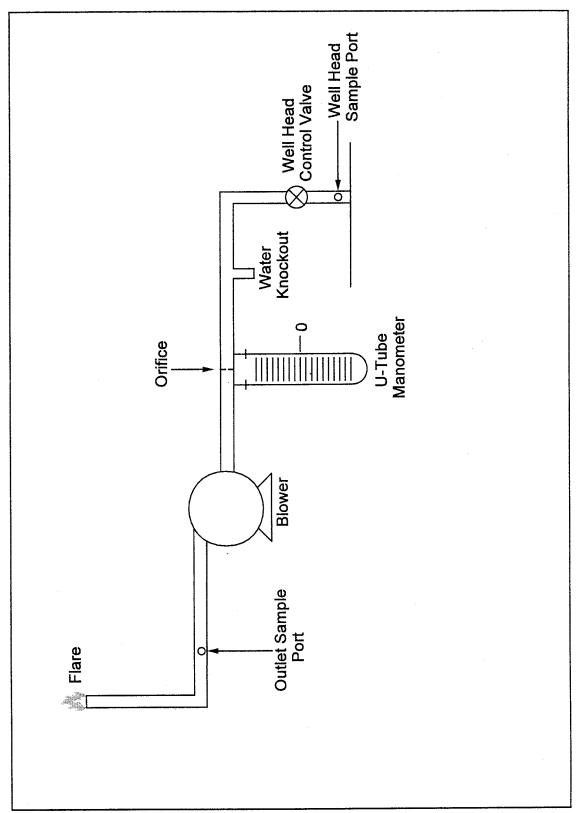


Figure 2E-1. Schematic of Aboveground Well Head Assembly.

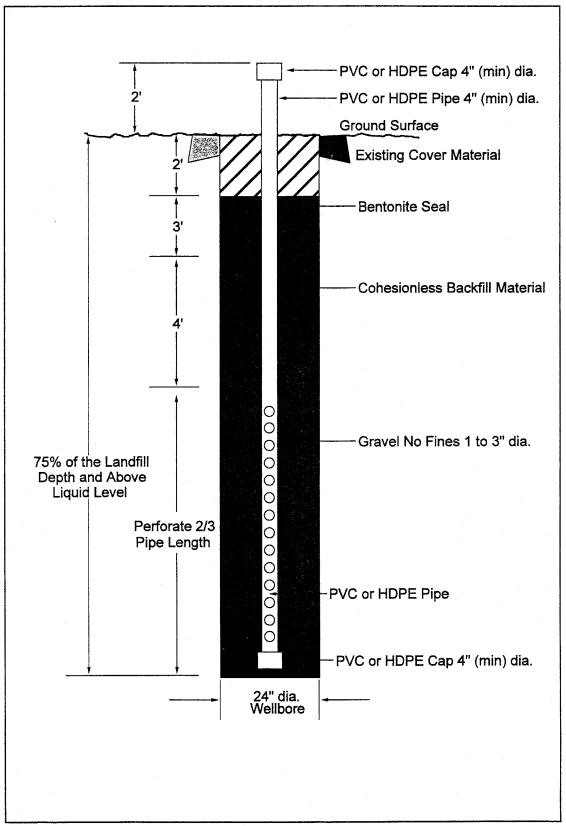


Figure 2E-3.

Gas Extraction Well.

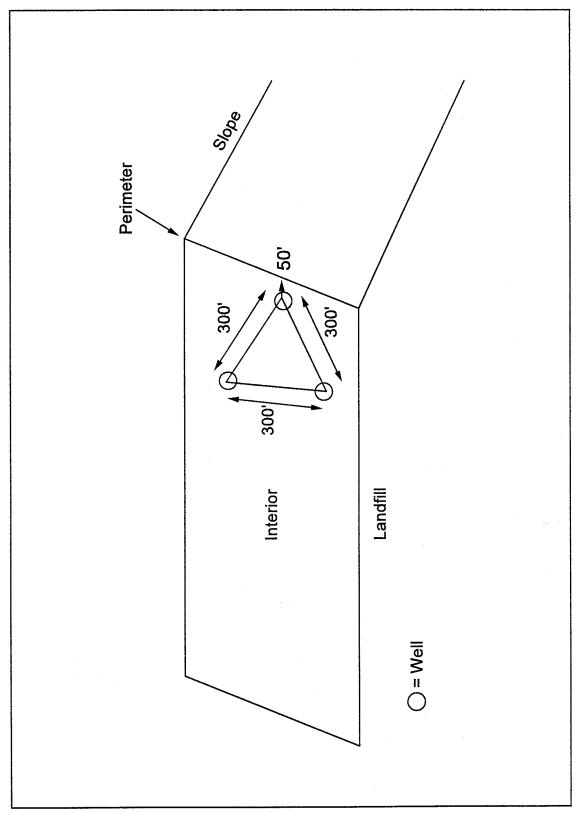


Figure 2E-2. Cluster Well Placement.

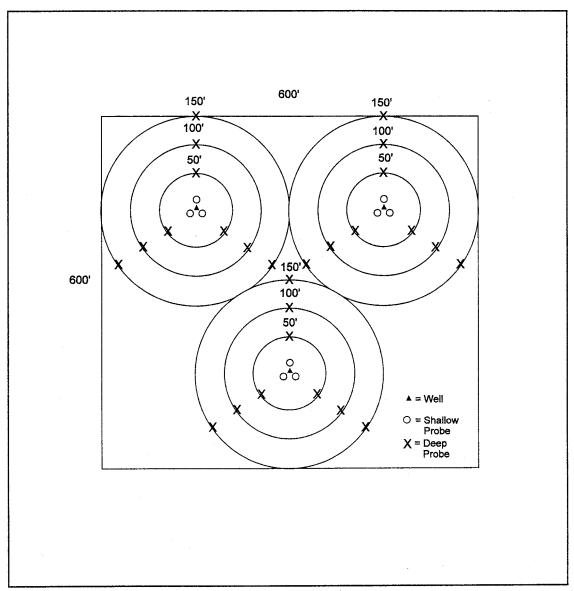


Figure 2E-4. Cluster Well Configuration.

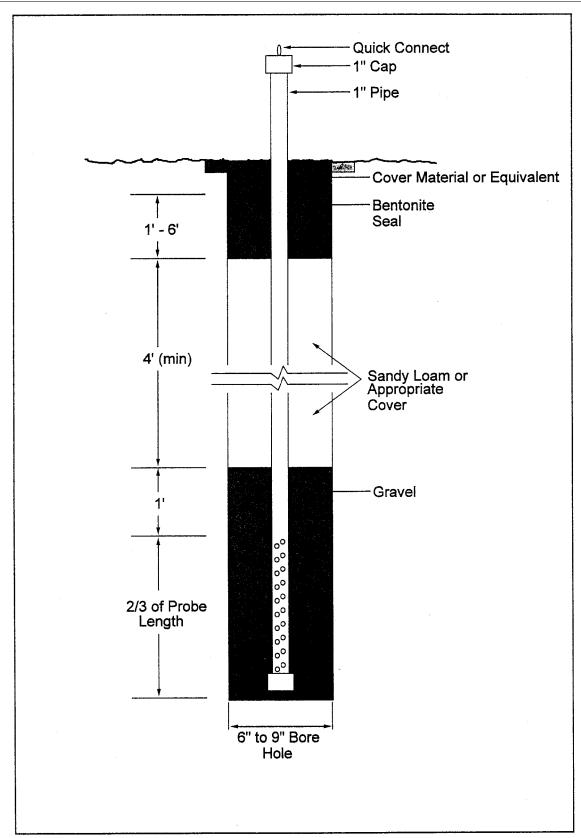


Figure 2E-5. Pressure Probe.

Method 3—Gas Analysis for the Determination of Dry Molecular Weight

Note: This method does not include all of the specifications (*e.g.*, equipment and

supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method

should also have a thorough knowledge of Method 1.

1.0 Scope and Application

1.1 Analytes.

Analytes	CAS No.	Sensitivity
Oxygen (O ₂) Nitrogen (N ₂) Carbon dioxide (CO ₂) Carbon monoxide (CO)	7727–37–9	2,000 ppmv.

- 1.2 Applicability. This method is applicable for the determination of CO_2 and O_2 concentrations and dry molecular weight of a sample from an effluent gas stream of a fossil-fuel combustion process or other process.
- 1.3 Other methods, as well as modifications to the procedure described herein, are also applicable for all of the above determinations. Examples of specific methods and modifications include: (1) A multi-point grab sampling method using an Orsat analyzer to analyze the individual grab sample obtained at each point; (2) a method for measuring either CO₂ or O₂ and using stoichiometric calculations to determine dry molecular weight; and (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator. The method may also be applicable to other processes where it has been determined that compounds other than CO₂, O₂, carbon monoxide (CO), and nitrogen (N₂) are not present in concentrations sufficient to affect the
- 1.4 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

- 2.1 A gas sample is extracted from a stack by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO_2 and percent O_2 . For dry molecular weight determination, either an Orsat or a Fyrite analyzer may be used for the analysis.
- 3.0 Definitions [Reserved]
- 4.0 Interferences
- 4.1 Several compounds can interfere, to varying degrees, with the results of Orsat or Fyrite analyses. Compounds that interfere with CO₂

concentration measurement include acid gases (e.g., sulfur dioxide, hydrogen chloride); compounds that interfere with O₂ concentration measurement include unsaturated hydrocarbons (e.g., acetone, acetylene), nitrous oxide, and ammonia. Ammonia reacts chemically with the O₂ absorbing solution, and when present in the effluent gas stream must be removed before analysis.

5.0 Safety

- 5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.
 - 5.2 Corrosive Reagents.
- 5.2.1 A typical Orsat analyzer requires four reagents: a gas-confining solution, CO_2 absorbent, O_2 absorbent, and CO absorbent. These reagents may contain potassium hydroxide, sodium hydroxide, cuprous chloride, cuprous sulfate, alkaline pyrogallic acid, and/or chromous chloride. Follow manufacturer's operating instructions and observe all warning labels for reagent use.
- 5.2.2 A typical Fyrite analyzer contains zinc chloride, hydrochloric acid, and either potassium hydroxide or chromous chloride. Follow manufacturer's operating instructions and observe all warning labels for reagent use.

6.0 Equipment and Supplies

Note: As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used, provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are, otherwise, capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

6.1 Grab Sampling (See Figure 3–1).

- 6.1.1 Probe. Stainless steel or borosilicate glass tubing equipped with an in-stack or out-of-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials, resistant to temperature at sampling conditions and inert to all components of the gas stream, may be used for the probe. Examples of such materials may include aluminum, copper, quartz glass, and Teflon.
- 6.1.2 Pump. A one-way squeeze bulb, or equivalent, to transport the gas sample to the analyzer.
- 6.2 Integrated Sampling (Figure 3–2).
- 6.2.1 Probe. Same as in Section 6.1.1.
- 6.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser no greater than 250 ml that will not remove O_2 , CO_2 , CO, and N_2 , to remove excess moisture which would interfere with the operation of the pump and flowmeter.
- 6.2.3 Valve. A needle valve, to adjust sample gas flow rate.
- 6.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rate meter.
- 6.2.5 Rate Meter. A rotameter, or equivalent, capable of measuring flow rate to \pm 2 percent of the selected flow rate. A flow rate range of 500 to 1000 ml/min is suggested.
- 6.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and duration of the test run. A capacity in the range of 55 to 90 liters (1.9 to 3.2 ft³) is suggested. To leak-check the bag, connect it to a water manometer, and pressurize the bag to 5 to 10 cm H₂O (2 to 4 in. H₂O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to

- 5 to 10 cm (2 to 4 in.) H_2O and allow to stand overnight. A deflated bag indicates a leak.
- 6.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 30 cm (12 in.), for the flexible bag leak-check.
- 6.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm (30 in.) Hg, for the sampling train leak-check.
- 6.3 Analysis. An Orsat or Fyrite type combustion gas analyzer.

7.0 Reagents and Standards

- 7.1 Reagents. As specified by the Orsat or Fyrite-type combustion analyzer manufacturer.
- 7.2 Standards. Two standard gas mixtures, traceable to National Institute of Standards and Technology (NIST) standards, to be used in auditing the accuracy of the analyzer and the analyzer operator technique:
- 7.2.1. Gas cylinder containing 2 to 4 percent O_2 and 14 to 18 percent O_2 .
- 7.2.2. Gas cylinder containing 2 to 4 percent CO_2 and about 15 percent O_2 .
- 8.0 Sample Collection, Preservation, Storage, and Transport
- 8.1 Single Point, Grab Sampling Procedure.
- 8.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.0 m (3.3 ft), unless otherwise specified by the Administrator.
- 8.1.2 Set up the equipment as shown in Figure 3–1, making sure all

- connections ahead of the analyzer are tight. If an Orsat analyzer is used, it is recommended that the analyzer be leakchecked by following the procedure in Section 11.5; however, the leak-check is optional.
- 8.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point. Purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer, and immediately analyze it for percent CO_2 and percent O_2 according to Section 11.2.
- 8.2 Single-Point, Integrated Sampling Procedure.
- 8.2.1 The sampling point in the duct shall be located as specified in Section 8.1.1.
- 8.2.2 Leak-check (optional) the flexible bag as in Section 6.2.6. Set up the equipment as shown in Figure 3–2. Just before sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe, and place it in the stack, with the tip of the probe positioned at the sampling point. Purge the sampling line. Next, connect the bag, and make sure that all connections are tight.
- 8.2.3 Sample Collection. Sample at a constant rate (±10 percent). The sampling run should be simultaneous with, and for the same total length of

- time as, the pollutant emission rate determination. Collection of at least 28 liters (1.0 ft³) of sample gas is recommended; however, smaller volumes may be collected, if desired.
- 8.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO₂ and percent O₂ using either an Orsat analyzer or a Fyrite type combustion gas analyzer according to Section 11.3.

Note: When using an Orsat analyzer, periodic Fyrite readings may be taken to verify/confirm the results obtained from the Orsat.

- 8.3 Multi-Point, Integrated Sampling Procedure.
- 8.3.1 Unless otherwise specified in an applicable regulation, or by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of 12 traverse points shall be used for all other cases. The traverse points shall be located according to Method 1.
- 8.3.2 Follow the procedures outlined in Sections 8.2.2 through 8.2.4, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in Figure 3–3.
- 9.0 Quality Control

Section	Quality control measure	Effect
8.2 10.1	Use of Fyrite to confirm Orsat results Periodic audit of analyzer and operator technique.	Ensures the accurate measurement of CO ₂ and O ₂ . Ensures that the analyzer is operating properly and that the operator performs the sampling procedure correctly and accurately.
11.3	Replicable analyses of integrated samples	Minimizes experimental error.

10.0 Calibration and Standardization

- 10.1 Analyzer. The analyzer and analyzer operator's technique should be audited periodically as follows: take a sample from a manifold containing a known mixture of CO_2 and O_2 , and analyze according to the procedure in Section 11.3. Repeat this procedure until the measured concentration of three consecutive samples agrees with the stated value \pm 0.5 percent. If necessary, take corrective action, as specified in the analyzer users manual.
- 10.2 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

11.0 Analytical Procedure

- 11.1 Maintenance. The Orsat or Fyrite-type analyzer should be maintained and operated according to the manufacturers specifications.
- 11.2 Grab Sample Analysis. Use either an Orsat analyzer or a Fyrite-type combustion gas analyzer to measure O_2 and CO_2 concentration for dry molecular weight determination, using procedures as specified in the analyzer user's manual. If an Orsat analyzer is used, it is recommended that the Orsat leak-check, described in Section 11.5, be performed before this determination; however, the check is optional. Calculate the dry molecular weight as indicated in Section 12.0. Repeat the
- sampling, analysis, and calculation procedures until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).
- 11.3 Integrated Sample Analysis. Use either an Orsat analyzer or a Fyrite-type combustion gas analyzer to measure O_2 and CO_2 concentration for dry molecular weight determination, using procedures as specified in the analyzer user's manual. If an Orsat analyzer is used, it is recommended that the Orsat leak-check, described in Section 11.5, be performed before this determination; however, the check is

optional. Calculate the dry molecular weight as indicated in Section 12.0. Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

- 11.4 Standardization. A periodic check of the reagents and of operator technique should be conducted at least once every three series of test runs as outlined in Section 10.1.
- 11.5 Leak-Check Procedure for Orsat Analyzer. Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is as follows:
- 11.5.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing, and then close the pipette stopcock.

- 11.5.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette, and then close the manifold stopcock.
- 11.5.3 Record the meniscus position. 11.5.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.
- 11.5.5 For the Orsat analyzer to pass the leak-check, two conditions must be met:
- 11.5.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.
- 11.5.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.
- 11.5.6 If the analyzer fails the leak-check procedure, check all rubber connections and stopcocks to determine whether they might be the cause of the leak. Disassemble, clean, and regrease any leaking stopcocks. Replace leaking rubber connections. After the analyzer is

reassembled, repeat the leak-check procedure.

12.0 Calculations and Data Analysis

12.1 Nomenclature.

 M_d = Dry molecular weight, g/g-mole (lb/lb-mole).

 $%CO_2 = Percent CO_2$ by volume, dry basis.

%O₂ = Percent O₂ by volume, dry basis.%CO = Percent CO by volume, dry basis.

 $%N_2$ = Percent N_2 by volume, dry basis. 0.280 = Molecular weight of N_2 or CO, divided by 100.

0.320 = Molecular weight of O_2 divided by 100.

0.440 = Molecular weight of CO_2 divided by 100.

12.2 Nitrogen, Carbon Monoxide Concentration. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent.

12.3 Dry Molecular Weight. Use Equation 3–1 to calculate the dry molecular weight of the stack gas.

 $M_d = 0.440 (\%CO_2) + 0.320 (\%O_2) + 0.280 (\%N_2 + \%CO)$ Eq. 3-1

Note: The above Equation 3–1 does not consider the effect on calculated dry molecular weight of argon in the effluent gas. The concentration of argon, with a molecular weight of 39.9, in ambient air is about 0.9 percent. A negative error of approximately 0.4 percent is introduced. The tester may choose to include argon in the analysis using procedures subject to approval of the Administrator.

- 13.0 Method Performance [Reserved]
- 14.0 Pollution Prevention [Reserved]
- 15.0 Waste Management [Reserved]
- 16.0 References
- 1. Altshuller, A.P. Storage of Gases and Vapors in Plastic Bags. International Journal of Air and Water Pollution. *6*:75–81. 1963.
- 2. Conner, William D. and J.S. Nader. Air Sampling with Plastic Bags. Journal of the American Industrial Hygiene Association. 25:291–297. 1964.
- 3. Burrell Manual for Gas Analysts, Seventh edition. Burrell Corporation, 2223 Fifth Avenue, Pittsburgh, PA. 15219. 1951.
- 4. Mitchell, W.J. and M.R. Midgett. Field Reliability of the Orsat Analyzer. Journal of Air Pollution Control Association. *26*:491– 495. May 1976.
- 5. Shigehara, R.T., R.M. Neulicht, and W.S. Smith. Validating Orsat Analysis Data from Fossil Fuel-Fired Units. Stack Sampling News. 4(2):21–26. August 1976.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

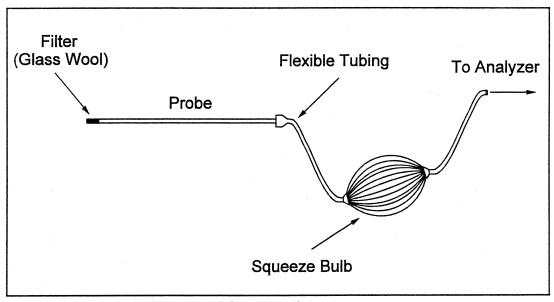


Figure 3-1. Grab-Sampling Train.

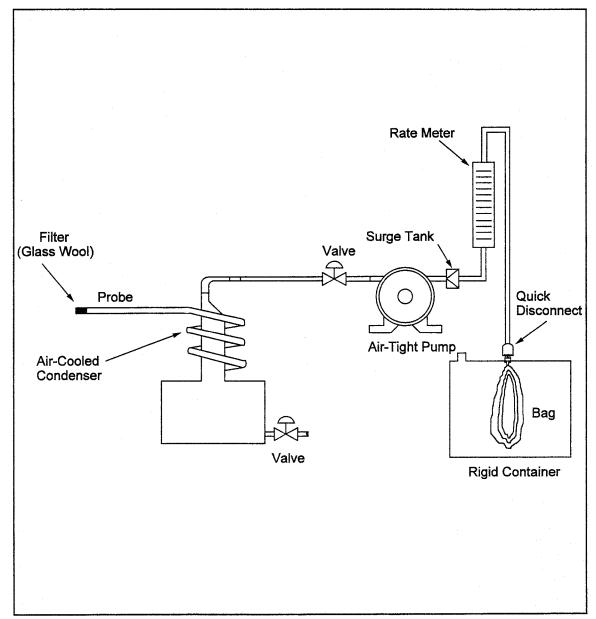


Figure 3-2. Integrated Gas-Sampling Train.

Time	Traverse point	Q (liter/min)	% Deviation ^a
Average			

 $^{^{\}rm a}\,\%$ Dev.=[(Q $-\,Q_{\rm avg})/Q_{\rm avg}]\!\!\times\!\!100$ (Must be $\leq\!\!\pm\!10\%$)

Figure 3–3. Sampling Rate Data

Method 3B—Gas Analysis for the Determination of Emission Rate Correction Factor or Excess Air

Note: This method does not include all of the specifications (*e.g.*, equipment and

supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least

the following additional test methods: Method 1 and 3.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Oxygen (O ₂)		2,000 ppmv. 2,000 ppmv. N/A.

- 1.2 Applicability. This method is applicable for the determination of O₂, CO₂, and CO concentrations in the effluent from fossil-fuel combustion processes for use in excess air or emission rate correction factor calculations. Where compounds other than CO₂, O₂, CO, and nitrogen (N₂) are present in concentrations sufficient to affect the results, the calculation procedures presented in this method must be modified, subject to the approval of the Administrator.
- 1.3 Other methods, as well as modifications to the procedure described herein, are also applicable for all of the above determinations. Examples of specific methods and modifications include: (1) A multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point, and (2) a method using CO₂ or O₂ and stoichiometric calculations to determine excess air. These methods and modifications may be used, but are subject to the approval of the Administrator.
- 1.4 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

- 2.1 A gas sample is extracted from a stack by one of the following methods: (1) Single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO₂, percent O₂, and, if necessary, percent CO using an Orsat combustion gas analyzer.
- 3.0 Definitions [Reserved]
- 4.0 Interferences
- 4.1 Several compounds can interfere, to varying degrees, with the results of Orsat analyses. Compounds that interfere with CO2 concentration measurement include acid gases (e.g., sulfur dioxide, hydrogen chloride);

compounds that interfere with O_2 concentration measurement include unsaturated hydrocarbons (e.g., acetone, acetylene), nitrous oxide, and ammonia. Ammonia reacts chemically with the O_2 absorbing solution, and when present in the effluent gas stream must be removed before analysis.

5.0 Safety

- 5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.
- 5.2 Corrosive Reagents. A typical Orsat analyzer requires four reagents: a gas-confining solution, CO₂ absorbent, O₂ absorbent, and CO absorbent. These reagents may contain potassium hydroxide, sodium hydroxide, cuprous chloride, cuprous sulfate, alkaline pyrogallic acid, and/or chromous chloride. Follow manufacturer's operating instructions and observe all warning labels for reagent use.

6.0 Equipment and Supplies

Note: As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used, provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are, otherwise, capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

- 6.1 Grab Sampling and Integrated Sampling. Same as in Sections 6.1 and 6.2, respectively for Method 3.
- 6.2 Analysis. An Orsat analyzer only. For low CO_2 (less than 4.0 percent) or high O_2 (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions. For Orsat maintenance and operation procedures, follow the instructions recommended by the

manufacturer, unless otherwise specified herein.

- 7.0 Reagents and Standards
- 7.1 Reagents. Same as in Method 3, Section 7.1.
- 7.2 Standards. Same as in Method 3, Section 7.2.
- 8.0 Sample Collection, Preservation, Storage, and Transport

Note: Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator. A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determinations, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight (see Method 3).

- 8.1 Single-Point, Grab Sampling and Analytical Procedure.
- 8.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.0 m (3.3 ft), unless otherwise specified by the Administrator.
- 8.1.2 Set up the equipment as shown in Figure 3–1 of Method 3, making sure all connections ahead of the analyzer are tight. Leak-check the Orsat analyzer according to the procedure described in Section 11.5 of Method 3. This leak-check is mandatory.
- 8.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer. For emission rate correction factor determinations, immediately analyze the sample for percent CO₂ or

percent O₂, as outlined in Section 11.2. For excess air determination, immediately analyze the sample for percent CO₂, O₂, and CO, as outlined in Section 11.2, and calculate excess air as outlined in Section 12.2.

- 8.1.4 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 11.5 of Method 3. For the results of the analysis to be valid, the Orsat analyzer must pass this leak-test before and after the analysis.
- 8.2 Single-Point, Integrated Sampling and Analytical Procedure.
- 8.2.1 The sampling point in the duct shall be located as specified in Section 8.1.1.
- 8.2.2 Leak-check (mandatory) the flexible bag as in Section 6.2.6 of Method 3. Set up the equipment as shown in Figure 3-2 of Method 3. Just before sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe, and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag, and make sure that all connections are tight.
- 8.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 28 liters (1.0 ft³) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.
- 8.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Section 11.2).
- 8.3 Multi-Point, Integrated Sampling and Analytical Procedure.
- 8.3.1 Unless otherwise specified in an applicable regulation, or by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of 12 traverse points shall be used for all other cases. The traverse points shall be located according to Method 1.

8.3.2 Follow the procedures outlined in Sections 8.2.2 through 8.2.4, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in Figure 3–3 of Method 3.

9.0 Quality Control

9.1 Data Validation Using Fuel Factor. Although in most instances, only CO_2 or O_2 measurement is required, it is recommended that both CO_2 and O_2 be measured to provide a check on the quality of the data. The data validation procedure of Section 12.3 is suggested.

Note: Since this method for validating the CO₂ and O₂ analyses is based on combustion of organic and fossil fuels and dilution of the gas stream with air, this method does not apply to sources that (1) remove CO₂ or O₂ through processes other than combustion, (2) add O_2 (e.g., oxygen enrichment) and N_2 in proportions different from that of air, (3) add CO₂ (e.g., cement or lime kilns), or (4) have no fuel factor, Fo, values obtainable (e.g., extremely variable waste mixtures). This method validates the measured proportions of CO₂ and O₂ for fuel type, but the method does not detect sample dilution resulting from leaks during or after sample collection. The method is applicable for samples collected downstream of most lime or limestone flue-gas desulfurization units as the CO₂ added or removed from the gas stream is not significant in relation to the total CO₂ concentration. The CO₂ concentrations from other types of scrubbers using only water or basic slurry can be significantly affected and would render the fuel factor check minimally useful.

10.0 Calibration and Standardization

10.1 Analyzer. The analyzer and analyzer operator technique should be audited periodically as follows: take a sample from a manifold containing a known mixture of CO_2 and O_2 , and analyze according to the procedure in Section 11.3. Repeat this procedure until the measured concentration of three consecutive samples agrees with the stated value ± 0.5 percent. If necessary, take corrective action, as specified in the analyzer users manual.

10.2 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

11.0 Analytical Procedure

- 11.1 Maintenance. The Orsat analyzer should be maintained according to the manufacturers specifications.
- 11.2 Grab Sample Analysis. To ensure complete absorption of the CO₂, O₂, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four)

should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.) Although in most cases, only CO_2 or O_2 concentration is required, it is recommended that both CO_2 and O_2 be measured, and that the procedure in Section 12.3 be used to validate the analytical data.

Note: Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis.

- 11.3 Integrated Sample Analysis. The Orsat analyzer must be leak-checked (see Section 11.5 of Method 3) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 11.3.1 through 11.3.3) for percent CO_2 , O_2 , and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2 , percent O_2 , and percent CO from 100 percent; and (3) calculate percent excess air, as outlined in Section 12.2.
- 11.3.1 To ensure complete absorption of the CO_2 , O_2 , or if applicable, CO, follow the procedure described in Section 11.2.

Note: Although in most instances only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that the procedures in Section 12.3 be used to validate the analytical data.

- 11.3.2 Repeat the analysis until the following criteria are met:
- 11.3.2.1 For percent CO_2 , repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average three acceptable values of percent CO_2 , and report the results to the nearest 0.2 percent.
- 11.3.2.2 For percent O_2 , repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2 , and report the results to the nearest 0.1 percent.
- 11.3.2.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO, and

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report the results to the nearest 0.1 percent.

11.3.3 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 11.5 of Method 3. For the results of the analysis to be valid, the Orsat analyzer must pass this leak-test before and after the analysis.

11.4 Standardization. A periodic check of the reagents and of operator technique should be conducted at least once every three series of test runs as indicated in Section 10.1.

12.0 Calculations and Data Analysis

12.1 Nomenclature. Same as Section 12.1 of Method 3 with the addition of the following:

%EA = Percent excess air.

$$0.264 = \text{Ratio of } O_2 \text{ to } N_2 \text{ in air, } v/v.$$

12.2 Percent Excess Air. Determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2 , percent CO, and percent O_2 from 100 percent. Calculate the percent excess air (if applicable) by substituting the appropriate values of percent O_2 , CO, and O_2 into Equation 3B–1.

$$%EA = \frac{\%O_2 - 0.5 \%CO}{0.264 \%N_2 - (\%O_2 - 0.5 \%CO)} \times 100$$
 Eq. 3B-1

Note: The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternative methods, subject to approval of the Administrator, are required.

12.3 Data Validation When Both CO₂ and O₂ Are Measured.

12.3.1 Fuel Factor, F_o. Calculate the fuel factor (if applicable) using Equation 3B-2:

$$F_0 = \frac{20.9 - \%O_2}{\%CO_2}$$
 Eq. 3B-2

Where:

 $\%O_2$ = Percent O_2 by volume, dry basis. $\%CO_2$ = Percent CO_2 by volume, dry basis.

20.9 = Percent O_2 by volume in ambient air.

If CO is present in quantities measurable by this method, adjust the O_2 and CO_2 values using Equations 3B–3 and 3B–4 before performing the calculation for F_o :

$$%CO_2(adj) = %CO_2 + %CO$$
 Eq. 3B-3

 $%O_2(adj) = %O_2 - 0.5 %CO$ Eq. 3B-4

%CO = Percent CO by volume, dry basis.

12.3.2 Compare the calculated $F_{\rm o}$ factor with the expected $F_{\rm o}$ values. Table 3B–1 in Section 17.0 may be used in establishing acceptable ranges for the

expected $F_{\rm o}$ if the fuel being burned is known. When fuels are burned in combinations, calculate the combined fuel $F_{\rm d}$ and $F_{\rm c}$ factors (as defined in Method 19, Section 12.2) according to the procedure in Method 19, Sections 12.2 and 12.3. Then calculate the $F_{\rm o}$ factor according to Equation 3B–5.

$$F_{\rm o} = \frac{0.209 \text{ F}_{\rm d}}{F_{\rm c}}$$
 Eq. 3B-5

12.3.3 Calculated Fo values, beyond the acceptable ranges shown in this table, should be investigated before accepting the test results. For example, the strength of the solutions in the gas analyzer and the analyzing technique should be checked by sampling and analyzing a known concentration, such as air; the fuel factor should be reviewed and verified. An acceptability range of ±12 percent is appropriate for the F_o factor of mixed fuels with variable fuel ratios. The level of the emission rate relative to the compliance level should be considered in determining if a retest is appropriate; i.e., if the measured emissions are much lower or much greater than the compliance limit, repetition of the test would not significantly change the compliance status of the source and would be unnecessarily time consuming and costly.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

Same as Method 3, Section 16.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 3B–1.—F_o FACTORS FOR SELECTED FUELS

Fuel type	F _o range
Coal:	
Anthracite and lignite	1.016-1.130
Bituminous	1.083-1.230
Oil:	
Distillate	1.260-1.413
Residual	1.210-1.370
Gas:	
Natural	1.600-1.836
Propane	1.434-1.586
Butane	1.405-1.553
Wood	1.000-1.120
Wood bark	1.003-1.130

Method 4—Determination of Moisture Content in Stack Gases

Note: This method does not include all the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1. Method 5. and Method 6.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Water vapor (H ₂ O)	7732–18–5	N/A

1.2 Applicability. This method is applicable for the determination of the moisture content of stack gas.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

2.2 The method contains two possible procedures: a reference method

and an approximation method.

- 2.2.1 The reference method is used for accurate determinations of moisture content (such as are needed to calculate emission data). The approximation method, provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content (e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc.) are also acceptable.
- $2.\dot{2}.2$ The reference method is often conducted simultaneously with a pollutant emission measurement run. When it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent. These calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, to be capable of yielding results within one percent H_2O of the reference method.

3.0 Definitions [Reserved]

4.0 Interferences

The moisture content of saturated gas streams or streams that contain water droplets, as measured by the reference method, may be positively biased. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to ±1 °C (2 °F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 8.1.1.1) during the reference method traverse, and calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) Using a psychrometric chart and making appropriate corrections if the stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation

of the process), alternative methods, subject to the approval of the Administrator, shall be used.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

- 6.1 Reference Method. A schematic of the sampling train used in this reference method is shown in Figure 4–1.
- 6.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation, and equipped with a filter, either in-stack (e.g., a plug of glass wool inserted into the end of the probe) or heated out-of-stack (e.g., as described in Method 5), to remove particulate matter. When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.
- 6.1.2 Condenser. Same as Method 5, Section 6.1.1.8.
- 6.1.3 Cooling System. An ice bath container, crushed ice, and water (or equivalent), to aid in condensing moisture.
- 6.1.4 Metering System. Same as in Method 5, Section 6.1.1.9, except do not use sampling systems designed for flow rates higher than 0.0283 m³/min (1.0 cfm). Other metering systems, capable of maintaining a constant sampling rate to within 10 percent and determining sample gas volume to within 2 percent, may be used, subject to the approval of the Administrator.
- 6.1.5 Barometer and Graduated Cylinder and/or Balance. Same as Method 5, Sections 6.1.2 and 6.2.5, respectively.
- 6.2. Approximation Method. A schematic of the sampling train used in this approximation method is shown in Figure 4–2.
 - 6.2.1 Probe. Same as Section 6.1.1.
- 6.2.2 Condenser. Two midget impingers, each with 30-ml capacity, or equivalent.
- 6.2.3 Cooling System. Ice bath container, crushed ice, and water, to aid in condensing moisture in impingers.
- 6.2.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

- 6.2.5 Valve. Needle valve, to regulate the sample gas flow rate.
- 6.2.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.
- 6.2.7 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume to within 2 percent, and calibrated over the range of flow rates and conditions actually encountered during sampling.
- 6.2.8 Rate Meter. Rotameter, or equivalent, to measure the flow range from 0 to 3 liters/min (0 to 0.11 cfm).
 - 6.2.9 Graduated Cylinder. 25-ml.
- 6.2.10 Barometer. Same as Method 5, Section 6.1.2.
- 6.2.11 Vacuum Gauge. At least 760-mm (30-in.) Hg gauge, to be used for the sampling leak check.
- 7.0 Reagents and Standards [Reserved]
- 8.0 Sample Collection, Preservation, Transport, and Storage
- 8.1 Reference Method. The following procedure is intended for a condenser system (such as the impinger system described in Section 6.1.1.8 of Method 5) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.
 - 8.1.1 Preliminary Determinations.
- 8.1.1.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.
- 8.1.1.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m³/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

8.1.2 Preparation of Sampling Train.
8.1.2.1 Place known volumes of water in the first two impingers; alternatively, transfer water into the first two impingers and record the weight of each impinger (plus water) to the nearest 0.5 g. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

8.1.2.2 Set up the sampling train as shown in Figure 4–1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of approximately 120 °C (248 °F), to prevent water condensation ahead of the condenser. Allow time for the temperatures to stabilize. Place crushed ice and water in the ice bath container.

8.1.3 Leak Check Procedures. It is recommended, but not required, that the volume metering system and sampling train be leak-checked as follows:

8.1.3.1 Metering System. Same as Method 5, Section 8.4.1.

8.1.3.2 Sampling Train. Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder), and pull a 380 mm (15 in.) Hg vacuum. A lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.020 cfm), whichever

is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

8.1.4 Sampling Train Operation. During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each run, record the data required on a data sheet similar to that shown in Figure 4–3. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point at least once during each time increment.

Note: When Method 4 is used concurrently with an isokinetic method (*e.g.*, Method 5) the sampling rate should be maintained at isokinetic conditions rather than 10 percent of constant rate.

8.1.4.1 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump, and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the silica gel outlet.

8.1.4.2 After collecting the sample, disconnect the probe from the first impinger (or from the filter holder), and conduct a leak check (mandatory) of the sampling train as described in Section 8.1.3.2. Record the leak rate. If the leakage rate exceeds the allowable rate,

either reject the test results or correct the sample volume as in Section 12.3 of Method 5.

8.2 Approximation Method.

Note: The approximation method described below is presented only as a suggested method (see Section 2.0).

8.2.1 Place exactly 5 ml water in each impinger. Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet. Then, plug the probe inlet and pull a vacuum of at least 250 mm (10 in.) Hg. Note the time rate of change of the dry gas meter dial; alternatively, a rotameter (0 to 40 ml/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable.

Note: Release the probe inlet plug slowly before turning off the pump.

8.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 liters/min (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as indicated by Figure 4–4.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
Section 8.1.1.4	Leak rate of the sampling system cannot exceed four percent of the average sampling rate or 0.00057 m ³ /min (0.20 cfm).	Ensures the accuracy of the volume of gas sampled. (Reference Method)
Section 8.2.1	Leak rate of the sampling system cannot exceed two percent of the average sampling rate.	Ensures the accuracy of the volume of gas sampled. (Approximation Method)

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Note: Maintain a laboratory log of all calibrations.

- 10.1 Reference Method. Calibrate the metering system, temperature sensors, and barometer according to Method 5, Sections 10.3, 10.5, and 10.6, respectively.
- 10.2 Approximation Method. Calibrate the metering system and the barometer according to Method 6, Section 10.1 and Method 5, Section 10.6, respectively.
- 11.0 Analytical Procedure
- 11.1 Reference Method. Measure the volume of the moisture condensed in

each of the impingers to the nearest ml. Alternatively, if the impingers were weighed prior to sampling, weigh the impingers after sampling and record the difference in weight to the nearest 0.5 g. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4–5), and calculate the moisture content, as described in Section 12.0.

11.2 Approximation Method. Combine the contents of the two impingers, and measure the volume to the nearest 0.5 ml.

12.0 Data Analysis and Calculations

Carry out the following calculations, retaining at least one extra significant

figure beyond that of the acquired data. Round off figures after final calculation.

12.1 Reference Method.

12.1.1 Nomenclature.

 $B_{\rm ws}$ = Proportion of water vapor, by volume, in the gas stream.

 M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

 $P_{\rm m}$ = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 (mm Hg)(m³)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft³)/(lbmole)(°R) for English units.

 T_m = Absolute temperature at meter, °K (°R).

 T_{std} = Standard absolute temperature, 293 °K (528 °R).

 V_f = Final volume of condenser water, ml.

 V_i = Initial volume, if any, of condenser water, ml.

 V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

 $V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

 $V_{\mathrm{wc(std)}}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

 $V_{wsg(std)} = Volume of water vapor collected in silica gel, corrected to standard conditions, scm (scf).$

 W_f = Final weight of silica gel or silica gel plus impinger, g.

W_i = Initial weight of silica gel or silica gel plus impinger, g.
 Y = Dry gas meter calibration factor.

Y = Dry gas meter calibration factor. ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).

12.1.6 Verification of Constant Sampling Rate. For each time increment, determine the ΔV_m . Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results, and repeat the run.

12.1.7 In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see Section 4.1), and another based upon the results of the impinger analysis. The lower of these two values of B_{ws} shall be considered correct.

12.2 Approximation Method. The approximation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content for the purpose of determining isokinetic sampling rate settings.

12.2.1 Nomenclature.

 ${
m B_{wm}}$ = Approximate proportion by volume of water vapor in the gas stream leaving the second impinger, 0.025.

 B_{ws} = Water vapor in the gas stream, proportion by volume.

 M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

 $\rho_{\rm w}$ = Density of water, 0.9982 g/ml (0.002201 lb/ml).

12.1.2 Volume of Water Vapor Condensed.

$$V_{wc(std)} = \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_W}$$
 Eq. 4-1
= $K_1(V_f - V_i)$

Where:

 $K_1 = 0.001333 \text{ m}^3/\text{ml}$ for metric units, = 0.04706 ft³/ml for English units.

12.1.3 Volume of Water Collected in Silica Gel

$$V_{wsg(std)} = \frac{\left(W_f - W_i\right) R T_{std}}{P_{std} M_W K_2}$$
 Eq. 4-2
= $K_3 (W_f - W_i)$

Where:

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} \qquad \text{Eq. 4-4}$$

 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 [(mm Hg)(m³)]/[(g-mole)(K)] for metric units and 21.85 [(in. Hg)(ft³)]/[(lb-mole)(°R)] for English units.

 T_m = Absolute temperature at meter, °K (°R).

 T_{std} = Standard absolute temperature, 293 °K (528 °R).

 $V_{\rm f}$ = Final volume of impinger contents, ml.

 V_i = Initial volume of impinger contents, ml.

 V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

 $V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).

 $V_{\mathrm{wc(std)}}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

Y = Dry gas meter calibration factor.

 $\rho_{\rm w} = {\rm Density~of~water,~0.09982~g/ml} \\ (0.002201~lb/ml).$

12.2.2 Volume of Water Vapor Collected.

$$V_{\text{wc(std)}} = \frac{\left(V_{\text{f}} - V_{\text{i}}\right) \rho_{\text{w}} R T_{\text{std}}}{P_{\text{std}} M_{\text{W}}}$$
 Eq. 4-5
= $K_5 \left(V_{\text{f}} - V_{\text{i}}\right)$

Where:

 $K_5 = 0.001333$ m³/ml for metric units, = 0.04706 ft³/ml for English units.

12.2.3 Sample Gas Volume.

 $K_2 = 1.0 \text{ g/g}$ for metric units,

= 453.6 g/lb for English units.

 $K_3 = 0.001335 \text{ m}^3/\text{g}$ for metric units, = 0.04715 ft³/g for English units.

12.1.4 Sample Gas Volume.

$$\begin{split} V_{m(std)} &= \frac{V_m \ Y \ P_m T_{std}}{P_{std} T_m} \qquad \text{Eq. 4-3} \\ &= K_4 Y \frac{V_m P_m}{T_m} \end{split}$$

Where:

 $K_4 = 0.3855$ °K/mm Hg for metric units, = 17.64 °R/in. Hg for English units.

Note: If the post-test leak rate (Section 8.1.4.2) exceeds the allowable rate, correct the value of Vm in Equation 4–3, as described in Section 12.3 of Method 5.

12.1.5 Moisture Content.

$$V_{m(std)} = \frac{V_m Y P_m T_{std}}{P_{std} T_m}$$
 Eq. 4-6
$$= K_6 Y \frac{V_m P_m}{T_m}$$

Where:

 K_6 = 0.3855 °K/mm Hg for metric units, = 17.64 °R/in. Hg for English units.

12.2.4 Approximate Moisture Content.

$$B_{ws} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + B_{wm} \quad Eq. 4-7$$

$$= \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + (0.025)$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

The procedure described in Method 5 for determining moisture content is acceptable as a reference method.

17.0 References

1. Air Pollution Engineering Manual (Second Edition). Danielson, J.A. (ed.). U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, NC. Publication No. AP–40. 1973.

2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, CA. November 1963.

3. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co. Los Angeles, CA. Bulletin WP-50. 1968.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

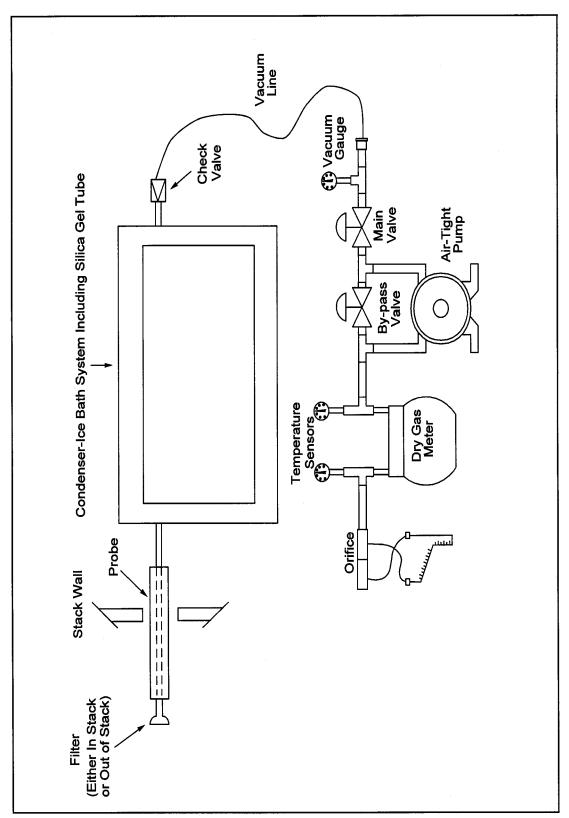


Figure 4-1. Moisture Sampling Train-Reference Method

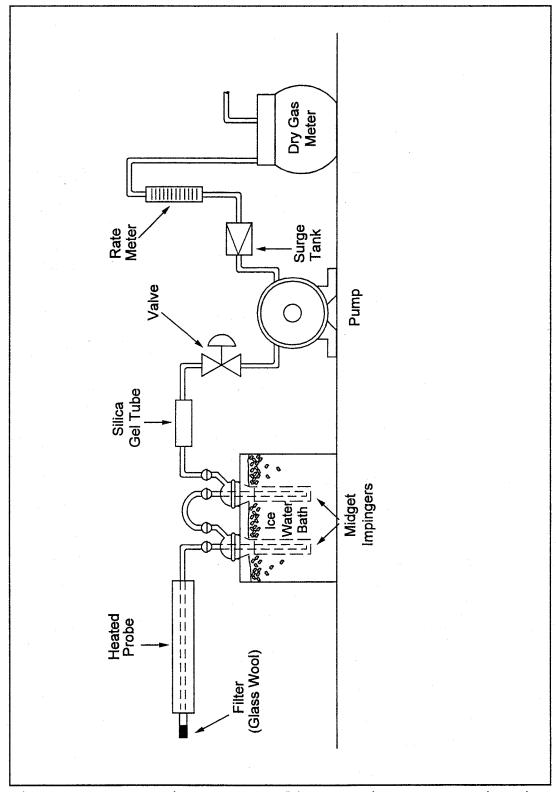


Figure 4-2. Moisture Sampling Train - Approximation Method.

Plant Location Operator Date Run No. Ambient temperature Barometric pressure Probe Length
Operator Date Run No.
Date Run No.
Run No.
Ambient temperature Barometric pressure Probe Length
Barometric pressure Probe Length
Probe Length

SCHEMATIC OF STACK CROSS SECTION

	Sampling	Stack	Pressure differential across ori-	Meter reading gas	ΔV_{m} m³ (ft³)	Gas sample temperature at dry gas meter		Tempera- ture of gas leaving
Traverse Pt. No.	time (Δ), min	temperature °C (°F)	fice meter ΔH mm (in.) H ₂ O	sample volume m³ (ft³)		Inlet Tm _{in} °C (°F)	Outlet Tm _{out} °C (°F)	condenser or last impinger °C (°F)
Average								

Location Test Date Operator Barometric pressure Comments:

Figure 4-3. Moisture Determination—Reference Method

Clock time	Gas Volume through meter, (V _m), m ³ (ft ³)	Rate meter setting m³/min (ft³/min)	Meter temperature °C (°F)

Figure 4–4. Example Moisture Determination Field Data Sheet—Approximation Method

	Impinger volume, ml	Silica gel weight, g
Final Initial Difference		

Figure 4-5. Analytical Data—Reference Method

Matter Emissions From Stationary Sources

Note: This method does not include all of the specifications (e.g., equipment and

 $\textbf{Method 5--} \textbf{Determination of Particulate} \quad \text{supplies) and procedures (\it{e.g.}, sampling and \textit{procedures}) and procedures (\it{e.g.}, sampling and sampl$ analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge

of at least the following additional test methods: Method 1, Method 2, Method 3.

- 1.0 Scope and Application
- 1.1 Analyte. Particulate matter (PM). No CAS number assigned.
- 1.2 Applicability. This method is applicable for the determination of PM emissions from stationary sources.
- 1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of $120\pm14^{\circ}C$ ($248\pm25^{\circ}F$) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. The PM mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water.

- 3.0 Definitions [Reserved]
- 4.0 Interferences [Reserved]
- 5.0 Safety
- 5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.
- 6.0 Equipment and Supplies
- 6.1 Sample Collection. The following items are required for sample collection:
- 6.1.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5–1 in Section 18.0. Complete construction details are given in APTD–0581 (Reference 2 in Section 17.0); commercial models of this train are also available. For changes from APTD–0581 and for allowable modifications of the train shown in Figure 5–1, see the following subsections.

Note: The operating and maintenance procedures for the sampling train are described in APTD-0576 (Reference 3 in Section 17.0). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein

6.1.1.1 Probe Nozzle. Stainless steel (316) or glass with a sharp, tapered

- leading edge. The angle of taper shall be ≤30°, and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used, subject to the approval of the Administrator. A range of nozzle sizes suitable for isokinetic sampling should be available. Typical nozzle sizes range from 0.32 to 1.27 cm (1/8 to 1/2 in) inside diameter (ID) in increments of 0.16 cm (1/16 in). Larger nozzles sizes are also available if higher volume sampling trains are used. Each nozzle shall be calibrated, according to the procedures outlined in Section 10.1.
- 6.1.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a probe gas temperature during sampling of 120 \pm 14 °C (248 \pm 25 °F), or such other temperature as specified by an applicable subpart of the standards or as approved by the Administrator for a particular application. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable. Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480 °C (900 °F); quartz glass liners shall be used for temperatures between 480 and 900 °C (900 and 1,650 °F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate glass is 820 °C (1500°F), and for quartz glass it is 1500 °C (2700 °F). Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825 or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.
- 6.1.1.3 Pitot Tube. Type S, as described in Section 6.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5–1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2–7) during sampling. The Type S pitot tube assembly shall

have a known coefficient, determined as outlined in Section 10.0 of Method 2.

6.1.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 6.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

6.1.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, or Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

6.1.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder of 120 ±14 °C (248 ±25 °F) during sampling, or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application.

6.1.1.7 Temperature Sensor. A temperature sensor capable of measuring temperature to within ± 3 °C (5.4 °F) shall be installed so that the sensing tip of the temperature sensor is in direct contact with the sample gas, and the temperature around the filter holder can be regulated and monitored

during sampling.
6.1.1.8 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leakfree ground glass fittings or any similar leak-free noncontaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) ID glass tube extending to about 1.3 cm ($\frac{1}{2}$ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 8.3.1), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A temperature sensor, capable of measuring temperature to within 1 °C (2 °F) shall be placed at the outlet of the fourth impinger for monitoring purposes. Alternatively, any system that cools the sample gas stream and allows

measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. An acceptable technique involves the measurement of condensed water either gravimetrically or volumetrically and the determination of the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20 °C (68 °F) and determining the weight gain. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

Note: If a determination of the PM collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

- 6.1.1.9 Metering System. Vacuum gauge, leak-free pump, temperature sensors capable of measuring temperature to within 3 °C (5.4 °F), dry gas meter (DGM) capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5–1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall allow periodic checks of isokinetic rates.
- 6.1.1.10 Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD–0581 or APTD–0576 may be used provided that the specifications of this method are met.
- 6.1.2 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in.).

Note: The barometric pressure reading may be obtained from a nearby National Weather Service station. In this case, the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be made at a rate of minus 2.5 mm Hg (0.1 in.) per 30 m (100 ft) elevation increase or plus 2.5 mm

Hg (0.1 in) per 30 m (100 ft) elevation decrease.

- 6.1.3 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 6.3 and 6.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2–4). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature sensor need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)
- 6.2 Sample Recovery. The following items are required for sample recovery:
- 6.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) constructed of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.
- 6.2.2 Wash Bottles. Two Glass wash bottles are recommended. Alternatively, polyethylene wash bottles may be used. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.
- 6.2.3 Glass Sample Storage
 Containers. Chemically resistant,
 borosilicate glass bottles, for acetone
 washes, 500 ml or 1000 ml. Screw cap
 liners shall either be rubber-backed
 Teflon or shall be constructed so as to
 be leak-free and resistant to chemical
 attack by acetone. (Narrow mouth glass
 bottles have been found to be less prone
 to leakage.) Alternatively, polyethylene
 bottles may be used.
- 6.2.4 Petri Dishes. For filter samples; glass or polyethylene, unless otherwise specified by the Administrator.
- 6.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml.
- 6.2.6 Plastic Storage Containers. Airtight containers to store silica gel.
- 6.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to

- container; not necessary if silica gel is weighed in the field.
- 6.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.
- 6.3 Sample Analysis. The following equipment is required for sample analysis:
 - 6.3.1 Glass Weighing Dishes.
 - 6.3.2 Desiccator.
- 6.3.3 Analytical Balance. To measure to within 0.1 mg.
- 6.3.4 Balance. To measure to within 0.5 g.
 - 6.3.5 Beakers. 250 ml.
- 6.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.
- 6.3.7 Temperature Sensor. To measure the temperature of the laboratory environment.
- 7.0 Reagents and Standards
- 7.1 Sample Collection. The following reagents are required for sample collection:
- 7.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Method D 2986-71, 78, or 95a (incorporated by reference—see § 60.17). Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO_2 or SO_3 , the filter material must be of a type that is unreactive to SO₂ or SO₃. Reference 10 in Section 17.0 may be used to select the appropriate filter.
- 7.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.
- 7.1.3 Water. When analysis of the material caught in the impingers is required, deionized distilled water (to conform to ASTM D 1193–77 or 91 Type 3 (incorporated by reference—see § 60.17)) shall be used. Run blanks prior to field use to eliminate a high blank on test samples.
 - 7.1.4 Crushed Ice.
- 7.1.5 Stopcock Grease. Acetoneinsoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.
- 7.2 Sample Recovery. Acetone, reagent grade, ≤0.001 percent residue, in glass bottles, is required. Acetone from metal containers generally has a high

residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

7.3 Sample Analysis. The following reagents are required for sample

analysis:

- 7.3.1 Acetone. Same as in Section 7.2.
- 7.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.
- 8.0 Sample Collection, Preservation, Storage, and Transport
- 8.1 Pretest Preparation. It is suggested that sampling equipment be maintained according to the procedures described in APTD-0576.
- 8.1.1 Place 200 to 300 g of silica gel in each of several air-tight containers. Weigh each container, including silica gel, to the nearest 0.5 g, and record this weight. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.
- 8.1.2 Check filters visually against light for irregularities, flaws, or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or polyethylene petri dishes), and keep each filter in its identified container at all times except

during sampling.

- 8.1.3 Desiccate the filters at 20 ± 5.6 $^{\circ}$ C (68 \pm 10 $^{\circ}$ F) and ambient pressure for at least 24 hours. Weigh each filter (or filter and shipping container) at intervals of at least 6 hours to a constant weight (i.e., ≤ 0.5 mg change from previous weighing). Record results to the nearest 0.1 mg. During each weighing, the period for which the filter is exposed to the laboratory atmosphere shall be less than 2 minutes. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105 °C (220 °F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.
- 8.2 Preliminary Determinations.8.2.1 Select the sampling site and the minimum number of sampling

- points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak check of the pitot lines (see Method 2, Section 8.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 8.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.
- 8.2.2 Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 8.3 of Method 2).
- 8.2.3 Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the required probe length.
- 8.2.4 Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (l) the sampling time per point is not less than 2 minutes (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.
- 8.2.5 The sampling time at each point shall be the same. It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.
- 8.2.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.
- 8.3 Preparation of Sampling Train.
 8.3.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to

- begin. Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.
- 8.3.2 Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.
- 8.3.3 When glass probe liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260 °C (500 °F) or a heatresistant string gasket when temperatures are higher. See APTD–0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as discussed above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each
- sampling point.
 8.3.4 Set up the train as shown in Figure 5–1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD–0576) to avoid the possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.
- 8.3.5 Place crushed ice around the impingers.
 - 8.4 Leak-Check Procedures.
- 8.4.1 Leak Check of Metering System Shown in Figure 5–1. That portion of the sampling train from the pump to the orifice meter should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5–2): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the

low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing, and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

8.4.2 Pretest Leak Check. A pretest leak check of the sampling train is recommended, but not required. If the pretest leak check is conducted, the following procedure should be used.

8.4.2.1 After the sampling train has been assembled, turn on and set the filter and probe heating systems to the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm (15 in.) Hg vacuum.

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

8.4.2.2 If a heat-resistant string is used, do not connect the probe to the train during the leak check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm (15 in.) Hg vacuum (see Note in Section 8.4.2.1). Then connect the probe to the train, and leak-check at approximately 25 mm (1 in.) Hg vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm (15 in.) Hg vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m³/ min (0.020 cfm), whichever is less, are unacceptable.

8.4.2.3 The following leak-check instructions for the sampling train described in APTD–0576 and APTD–0581 may be helpful. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve, and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve, as this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum, or end the leak check and start over.

8.4.2.4 When the leak check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable), and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and the silica gel from

being entrained backward into the third impinger.

8.4.3 Leak Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak check shall be conducted immediately before the change is made. The leak check shall be done according to the procedure outlined in Section 8.4.2 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m³/ min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, either record the leakage rate and plan to correct the sample volume as shown in Section 12.3 of this method, or void the sample run.

Note: Immediately after component changes, leak checks are optional. If such leak checks are done, the procedure outlined in Section 8.4.2 above should be used.

8.4.4 Post-Test Leak Check. A leak check of the sampling train is mandatory at the conclusion of each sampling run. The leak check shall be performed in accordance with the procedures outlined in Section 8.4.2, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m³ min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, either record the leakage rate and correct the sample volume as shown in Section 12.3 of this method, or void the sampling run.

8.5 Sampling Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120 \pm 14 °C (248 \pm 25 °F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

8.5.1 For each run, record the data required on a data sheet such as the one shown in Figure 5–3. Be sure to record the initial DGM reading. Record the DGM readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before

and after each leak check, and when sampling is halted. Take other readings indicated by Figure 5–3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

8.5.2 Clean the portholes prior to the test run to minimize the chance of collecting deposited material. To begin sampling, verify that the filter and probe heating systems are up to temperature, remove the nozzle cap, verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump, and adjust the flow to isokinetic conditions. Nomographs are available which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient (C_p) is 0.85 \pm 0.02, and the stack gas equivalent density [dry molecular weight (M_d)] is equal to 29 ± 4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Reference 7 in Section 17.0) are taken to compensate for the deviations.

8.5.3 When the stack is under significant negative pressure (*i.e.*, height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

8.5.4 When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

8.5.5 Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.

8.5.6 During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the condenser/silica gel outlet. Also,

periodically check the level and zero of the manometer.

8.5.7 If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of the sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check (see Section 8.4.3). The total PM weight shall include the summation of the filter assembly catches.

8.5.8 A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note: When two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

- 8.5.9 At the end of the sample run, close the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final DGM meter reading, and conduct a post-test leak check, as outlined in Section 8.4.4. Also, leak-check the pitot lines as described in Method 2, Section 8.1. The lines must pass this leak check, in order to validate the velocity head data.
- 8.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, Section 12.11) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates because of source conditions, consult with the Administrator for possible variance on the isokinetic rates.
 - 8.7 Sample Recovery.
- 8.7.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.
- 8.7.2 When the probe can be safely handled, wipe off all external PM near the tip of the probe nozzle, and place a cap over it to prevent losing or gaining PM. Do not cap off the probe tip tightly while the sampling train is cooling down. This would create a vacuum in

the filter holder, thereby drawing water from the impingers into the filter holder.

- 8.7.3 Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened, and cap it. Remove the umbilical cord from the last impinger, and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder, and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.
- 8.7.4 Transfer the probe and filterimpinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.
- 8.7.5 Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used, and place it in a glass sample container labeled "acetone blank."
- 8.7.6 Inspect the train prior to and during disassembly, and note any abnormal conditions. Treat the samples as follows:
- 8.7.6.1 Container No. 1. Carefully remove the filter from the filter holder, and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the PM cake is inside the fold. Using a dry Nylon bristle brush and/or a sharp-edged blade, carefully transfer to the petri dish any PM and/or filter fibers that adhere to the filter holder gasket. Seal the container.
- 8.7.6.2 Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover PM or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Deionized distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator. In these cases, save a water blank, and follow the Administrator's directions on analysis. Perform the acetone rinse as follows:

- 8.7.6.2.1 Carefully remove the probe nozzle. Clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.
- 8.7.6.2.2 Brush and rinse the inside parts of the fitting with acetone in a similar way until no visible particles remain.
- 8.7.6.2.3 Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter that is brushed from the probe. Run the brush through the probe three times or more until no visible PM is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe.
- 8.7.6.2.4 It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.
- 8.7.6.2.5 After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to allow determination of whether leakage

occurred during transport. Label the container to identify clearly its contents.

8.7.6.3 Container No. 3. Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container, and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to

be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 in Section 11.2.3.

8.7.6.4 Impinger Water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid that is in the first three impingers to within 1 ml by using a graduated cylinder or by weighing it to within 0.5 g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after

measuring and recording the volume or weight, unless analysis of the impinger catch is required (see NOTE, Section 6.1.1.8). If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

8.8 Sample Transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect		
8.4, 10.1–10.6	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.		

9.2 Volume Metering System Checks. The following procedures are suggested to check the volume metering system calibration values at the field test site prior to sample collection. These procedures are optional.

9.2.1 Meter Orifice Check. Using the calibration data obtained during the calibration procedure described in Section 10.3, determine the $\Delta H@$ for the metering system orifice. The $\Delta H@$ is the orifice pressure differential in units of in. H_2O that correlates to 0.75 cfm of air at 528 °R and 29.92 in. Hg. The $\Delta H@$ is calculated as follows:

$$\Delta H @= 0.0319 \ \Delta H \frac{T_M \theta^2}{P_{har} Y^2 V_m^2}$$

Where:

 ΔH = Average pressure differential across the orifice meter, in. H_2O .

 $T_{\rm m}$ = Absolute average DGM temperature, °R.

 P_{bar} = Barometric pressure, in. Hg.

 θ = Total sampling time, min.

Y = DGM calibration factor, dimensionless.

 $V_{\rm m}$ = Volume of gas sample as measured by DGM, dcf.

 $0.0319 = (0.0567 \text{ in. Hg/}^{\circ}\text{R}) (0.75 \text{ cfm})^{2}$

9.2.1.1 Before beginning the field test (a set of three runs usually constitutes a field test), operate the metering system (i.e., pump, volume meter, and orifice) at the $\Delta H@$ pressure differential for 10 minutes. Record the volume collected, the DGM temperature, and the barometric pressure. Calculate a DGM calibration check value, Y_c , as follows:

$$Y_{c} = \frac{10}{V_{m}} \left[\frac{0.0319 \text{ T}_{m}}{P_{har}} \right]^{\frac{1}{2}}$$

where:

Y_c = DGM calibration check value, dimensionless.

10 = Run time, min.

9.2.1.2 Compare the Y_c value with the dry gas meter calibration factor Y to determine that: 0.97Y < Y_c < 1.03Y. If the Y_c value is not within this range, the volume metering system should be investigated before beginning the test.

9.2.2 Calibrated Critical Orifice. A critical orifice, calibrated against a wet test meter or spirometer and designed to be inserted at the inlet of the sampling meter box, may be used as a check by following the procedure of Section 16.2.

10.0 Calibration and Standardization

Note: Maintain a laboratory log of all calibrations.

10.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the ID of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

10.2 Pitot Tube Assembly. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 10.1 of Method 2.

10.3 Metering System.

10.3.1 Calibration Prior to Use. Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter that is accurate to within 1 percent. Refer to Figure 5-4. The wet test meter should have a capacity of 30 liters/rev (1 ft³/ rev). A spirometer of 400 liters (14 ft³) or more capacity, or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading as expected in field use to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the DGM. Also note the barometric pressure and the temperatures of the wet test meter, the inlet of the DGM, and the outlet of the DGM. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of 0.14 m³ (5 ft³) at all orifice settings. Record all the data on a form similar to Figure 5-5 and calculate Y, the DGM calibration factor, and $\Delta H_{@}$, the orifice calibration factor, at each orifice setting as shown on Figure 5-5. Allowable tolerances for

individual Y and $\Delta H_{@}$ values are given in Figure 5–5. Use the average of the Y values in the calculations in Section 12.0.

10.3.1.1 Before calibrating the metering system, it is suggested that a leak check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m³/min (0.020 cfm). At the end of the run, take the difference of the measured wet test meter and DGM volumes. Divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m³/min (0.020 cfm).

10.3.2 Calibration After Use. After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the DGM calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as detailed in Section 10.3.1.

Note: Alternative procedures (*e.g.*, rechecking the orifice meter coefficient) may be used, subject to the approval of the Administrator.

10.3.3 Acceptable Variation in Calibration. If the DGM coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (*i.e.*, before or after) gives the lower value of total sample volume.

10.4 Probe Heater Calibration. Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical sample flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD-0576 can also be used. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

Note: The probe heating system shall be calibrated before its initial use in the field.

10.5 Temperature Sensors. Use the procedure in Section 10.3 of Method 2 to calibrate in-stack temperature sensors. Dial thermometers, such as are used for the DGM and condenser outlet, shall be calibrated against mercury-inglass thermometers.

10.6 Barometer. Calibrate against a mercury barometer.

11.0 Analytical Procedure

- 11.1 Record the data required on a sheet such as the one shown in Figure 5–6.
- 11.2 Handle each sample container as follows:

11.2.1 Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose PM from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight, and report the results to the nearest 0.1 mg. For the purposes of this section, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings. Alternatively, the sample may be oven dried at 104 °C (220 °F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The sample may be oven dried at 104 °C (220 °F) for 2 to 3 hours. Once the sample has cooled, weigh the sample, and use this weight as a final weight.

11.2.2 Container No. 2. Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest

11.2.3 Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

11.2.4 Acetone Blank Container. Measure the acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Note: The contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used, provided that they give equivalent results.

12.1 Nomenclature.

 A_n = Cross-sectional area of nozzle, m^2 (ft²).

 B_{ws} = Water vapor in the gas stream, proportion by volume.

C_a = Acetone blank residue concentration, mg/mg.

c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (gr/ dscf).

I = Percent of isokinetic sampling.

 L_1 = Individual leakage rate observed during the leak-check conducted prior to the first component change, m^3/min (ft³/min)

- $L_{\rm a} = {\rm Maximum~acceptable~leakage~rate} \\ {\rm for~either~a~pretest~leak\text{-}check~or~for} \\ {\rm a~leak\text{-}check~following~a~component} \\ {\rm change;~equal~to~0.00057~m^3/min} \\ {\rm (0.020~cfm)~or~4~percent~of~the} \\ {\rm average~sampling~rate,~whichever~is} \\ {\rm less.} \\ \\$
- $\begin{aligned} L_i &= \text{Individual leakage rate observed} \\ &\text{during the leak-check conducted} \\ &\text{prior to the "ith" component change} \\ &\text{($i=1,2,3\dots n$), $m^3/min (cfm)$.} \end{aligned}$

 L_p = Leakage rate observed during the post-test leak-check, m³/min (cfm).

m_a = Mass of residue of acetone after evaporation, mg.

 m_n = Total amount of particulate matter collected, mg.

 M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

 P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

 P_s = Absolute stack gas pressure, mm Hg (in. Hg).

 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

 $R = Ideal \ gas \ constant, \ 0.06236 \ ((mm \ Hg)(m \ ^3))/((K)(g-mole)) \ \{21.85 \ ((in. \ Hg) \ (ft \ ^3))/((^\circR) \ (lb-mole))\}.$

T_m = Absolute average DGM temperature (see Figure 5-3), K (°R).

T_s = Absolute average stack gas temperature (see Figure 5-3), K (°R).

 T_{std} = Standard absolute temperature, 293 K (528 °R).

 V_a = Volume of acetone blank, ml.

 V_{aw} = Volume of acetone used in wash,

 V_{1c} = Total volume of liquid collected in impingers and silica gel (see Figure 5–6), ml.

 $V_{\rm m}$ = Volume of gas sample as measured

by dry gas meter, dcm (dcf). $V_{m(std)} = V$ olume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

 $V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).

 V_s = Stack gas velocity, calculated by Method 2, Equation 2-7, using data obtained from Method 5, m/sec (ft/

W_a = Weight of residue in acetone wash,

Y = Dry gas meter calibration factor.

 $\Delta H = Average pressure differential$ across the orifice meter (see Figure 5-4), mm H₂O (in. H₂O).

 ρ_a = Density of acetone, mg/ml (see label on bottle).

= Density of water, 0.9982 g/ ml.(0.002201 lb/ml).

 θ = Total sampling time, min.

 θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.

 θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.

 θ_p = Sampling time interval, from the final (n th) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5-3).

12.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20 °C, 760 mm Hg or 68 °F, 29.92 in. Hg) by using Equation 5–1.

$$V_{m(std)} = V_m Y \frac{T_{std} \left(P_{bar} + \frac{\Delta H}{13.6} \right)}{T_m P_{std}}$$
 Eq. 5-1
$$= K_1 V_m Y \frac{P_{bar} + \left(\frac{\Delta H}{13.6} \right)}{T_m}$$

Where:

 $K_1 = 0.3858$ °K/mm Hg for metric units, = 17.64 °R/in. Hg for English units.

Note: Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the posttest leak check or leak checks conducted prior to component changes) exceeds La. If Lp

or Li exceeds La, Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5-1 with the expression:

$$\left(V_{m}-\left(L_{p}-L_{a}\right)\theta\right)$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5–1 by the expression:

$$\left| V_m - (L_1 - L_a)\theta_1 - \sum_{i=2}^n (L_1 - L_a)\theta_i - (L_p - L_a)\theta_p \right|$$

and substitute only for those leakage rates (Li or Lp) which exceed La.

12.4 Volume of Water Vapor Condensed.

$$V_{w(std)} = V_{lc} \frac{\rho_w R T_{std}}{M_w P_{std}}$$
 Eq. 5-2
= $K_2 V_{lc}$

Where:

 $K_2 = 0.001333$ m³/ml for metric units, = 0.04706 ft 3/ml for English units. 12.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$
 Eq. 5-3

Note: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of Bws shall be considered correct. The procedure for determining the moisture content based upon the assumption of saturated conditions is given in Section 4.0 of Method 4. For the purposes of this method, the average stack gas temperature

from Figure 5-3 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is \pm 1°C (2°F).

12.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a} \qquad \text{Eq. 5-4}$$

12.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a$$
 Eq. 5-5

12.8 Total Particulate Weight. Determine the total particulate matter catch from the sum of the weights obtained from Containers 1 and 2 less the acetone blank (see Figure 5–6).

Note: In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight. Refer to Section 8.5.8 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

12.9 Particulate Concentration.

$$C_s = \frac{K_3 m_n}{V_{m(std)}}$$
 Eq. 5-6

Where:

 K_3 = 0.001 g/mg for metric units. = 0.0154 gr/mg for English units. 12.10 Conversion Factors:

From	То	Multiply by
ft ³	m ³ mg mg/m ³ g lb	0.02832 64.80004 2288.4 0.001 1.429 × 10 ⁻⁴

12.11 Isokinetic Variation.

12.11.1 Calculation from Raw Data.

$$I = \frac{100 \, T_s \left[K_4 V_{1c} + \frac{\left(V_m Y \right)}{T_m} \left(P_{bar} + \frac{\Delta H}{13.6} \right) \right]}{60 \, \theta v_s P_s A_n}$$

Where:

 $K_4 = 0.003454 \text{ ((mm Hg)(m}^3))/((ml)(^\circ\text{K}))$ for metric units.

= 0.002669 ((in. Hg)(ft³))/((ml)($^{\circ}$ R)) for English units.

12.11.2 Calculation from Intermediate Values.

$$\begin{split} I &= \frac{T_{s} V_{m(std)} P_{std} 100}{T_{std} v_{s} \theta A_{n} P_{s} 60 (1 - B_{ws})} \\ &= K_{5} \frac{T_{s} V_{m(std)}}{P_{s} v_{s} A_{n} \theta (1 - B_{ws})} \end{split}$$
 Eq. 5-8

Where:

 $K_5 = 4.320$ for metric units, = 0.09450 for English units.

12.11.3 Acceptable Results. If 90 percent $\leq I \leq 110$ percent, the results are acceptable. If the PM results are low in comparison to the standard, and "I" is over 110 percent or less than 90 percent, the Administrator may opt to accept the results. Reference 4 in Section 17.0 may be used to make acceptability judgments. If "I" is judged to be unacceptable, reject the results, and repeat the sampling run.

12.12 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in Sections 12.3 and 12.4 of Method 2.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 Dry Gas Meter as a Calibration Standard. A DGM may be used as a calibration standard for volume measurements in place of the wet test meter specified in Section 10.3, provided that it is calibrated initially and recalibrated periodically as follows:

16.1.1 Standard Dry Gas Meter Calibration.

16.1.1.1. The DGM to be calibrated and used as a secondary reference meter should be of high quality and have an appropriately sized capacity (e.g., 3 liters/rev (0.1 ft³/rev)). A spirometer (400 liters (14 ft³) or more capacity), or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should have a capacity of 30 liters/rev (1 ft3/rev) and capable of measuring volume to within 1.0 percent. Wet test meters should be checked against a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained.

16.1.1.2 Set up the components as shown in Figure 5–7. A spirometer, or equivalent, may be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test meter. The pressure drop indicated by the manometer at the inlet side of the DGM should be minimized (no greater than 100 mm $\rm H_2O$ (4 in. $\rm H_2O$) at a flow rate of 30 liters/min (1 cfm)). This can be accomplished by using large diameter tubing connections and straight pipe fittings.

16.1.1.3 Collect the data as shown in the example data sheet (see Figure 5–8). Make triplicate runs at each of the flow rates and at no less than five different flow rates. The range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm) or over the expected operating range.

16.1.1.4 Calculate flow rate, Q, for each run using the wet test meter volume, V_W , and the run time, θ . Calculate the DGM coefficient, Y_{ds} , for each run. These calculations are as follows:

$$Q = K_1 \frac{P_{bar} V_w}{\left(T_w + T_{std}\right)\theta}$$
 Eq. 5-9

$$Y_{ds} = \frac{V_{w} (T_{ds} + T_{std}) P_{bar}}{V_{ds} (T_{w} + T_{std}) (P_{bar} + \frac{\Delta p}{13.6})}$$
 Eq. 5-10

Where:

 $K_1 = 0.3858$ °C/mm Hg for metric units=17.64 °F/in. Hg for English units.

 V_W = Wet test meter volume, liter (ft³).

 V_{ds} = Dry gas meter volume, liter (ft³).

 T_{ds} = Average dry gas meter temperature, °C (°F).

 T_{adj} = 273 °C for metric units = 460 °F for English units.

 T_W = Average wet test meter temperature, °C (°F) P_{bar} = Barometric pressure, mm Hg (in. Hg).

 $\Delta p = Dry$ gas meter inlet differential pressure, mm H_2O (in. H_2O). $\theta = Run$ time, min.

16.1.1.5 Compare the three Y_{ds} values at each of the flow rates and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra sets of triplicate runs may be made in order to complete this requirement. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications cannot be met in three sets of successive triplicate runs, the meter is not suitable as a calibration standard and should not be used as such. If these specifications are met, average the three Yds values at each flow rate resulting in no less than five average meter coefficients, Y_{ds}.

 $16.\overline{1}.1.6$ Prepare a curve of meter coefficient, Y_{ds} , versus flow rate, Q, for the DGM. This curve shall be used as a reference when the meter is used to calibrate other DGMs and to determine whether recalibration is required.

16.1.2 Standard Dry Gas Meter Recalibration.

16.1.2.1 Recalibrate the standard DGM against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard DGM is kept in a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

16.1.2.2 As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates [suggested rates are 14 and 30 liters/min (0.5 and 1.0 cfm)]. Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within 1.5 percent of the calibration curve values at the same flow rates, the meter need not be recalibrated until the next date for a recalibration check.

16.2 Critical Orifices As Calibration Standards. Critical orifices may be used

as calibration standards in place of the wet test meter specified in Section 16.1, provided that they are selected, calibrated, and used as follows:

16.2.1 Selection of Critical Orifices. 16.2.1.1 The procedure that follows describes the use of hypodermic needles or stainless steel needle tubings which have been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices (i.e., a critical vacuum can be obtained, as described in Section 16.2.2.2.3). Select five critical orifices that are appropriately sized to cover the range of flow rates between 10 and 34 liters/min (0.35 and 1.2 cfm) or the expected operating range. Two of the critical orifices should bracket the expected operating range. A minimum of three critical orifices will be needed to calibrate a Method 5 DGM; the other two critical orifices can serve as spares and provide better selection for bracketing the range of operating flow rates. The needle sizes and tubing lengths shown in Table 5-1 in Section 18.0 give the approximate flow rates.

16.2.1.2 These needles can be adapted to a Method 5 type sampling train as follows: Insert a serum bottle stopper, 13 by 20 mm sleeve type, into a ½-inch Swagelok (or equivalent) quick connect. Insert the needle into the stopper as shown in Figure 5–9.

16.2.2 Critical Orifice Calibration. The procedure described in this section uses the Method 5 meter box configuration with a DGM as described in Section 6.1.1.9 to calibrate the critical orifices. Other schemes may be used, subject to the approval of the Administrator.

16.2.2.1 Calibration of Meter Box. The critical orifices must be calibrated in the same configuration as they will be used (*i.e.*, there should be no connections to the inlet of the orifice).

16.2.2.1.1 Before calibrating the meter box, leak check the system as follows: Fully open the coarse adjust valve, and completely close the by-pass valve. Plug the inlet. Then turn on the pump, and determine whether there is any leakage. The leakage rate shall be zero (*i.e.*, no detectable movement of the DGM dial shall be seen for 1 minute).

16.2.2.1.2 Check also for leakages in that portion of the sampling train

between the pump and the orifice meter. See Section 8.4.1 for the procedure; make any corrections, if necessary. If leakage is detected, check for cracked gaskets, loose fittings, worn O-rings, etc., and make the necessary repairs.

16.2.2.1.3 After determining that the meter box is leakless, calibrate the meter box according to the procedure given in Section 10.3. Make sure that the wet test meter meets the requirements stated in Section 16.1.1.1. Check the water level in the wet test meter. Record the DGM calibration factor, Y.

16.2.2.2 Calibration of Critical Orifices. Set up the apparatus as shown in Figure 5–10.

16.2.2.2.1 Allow a warm-up time of 15 minutes. This step is important to equilibrate the temperature conditions through the DGM.

16.2.2.2.2 Leak check the system as in Section 16.2.2.1.1. The leakage rate shall be zero.

16.2.2.2.3 Before calibrating the critical orifice, determine its suitability and the appropriate operating vacuum as follows: Turn on the pump, fully open the coarse adjust valve, and adjust the by-pass valve to give a vacuum reading corresponding to about half of atmospheric pressure. Observe the meter box orifice manometer reading, AH. Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer. Record the critical vacuum for each orifice. Orifices that do not reach a critical value shall not be used.

16.2.2.2.4 Obtain the barometric pressure using a barometer as described in Section 6.1.2. Record the barometric pressure, P_{bar} , in mm Hg (in. Hg).

16.2.2.2.5 Conduct duplicate runs at a vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. The runs shall be at least 5 minutes each. The DGM volume readings shall be in increments of complete revolutions of the DGM. As a guideline, the times should not differ by more than 3.0 seconds (this includes allowance for changes in the DGM temperatures) to achieve \pm 0.5 percent in K' (see Eq. 5–11). Record the information listed in Figure 5–11.

16.2.2.2.6 Calculate K' using Equation 5–11.

$$K^{1} = \frac{K_{1}V_{m}Y\left(P_{bar} + \frac{\Delta H}{13.6}\right)T_{amb}^{1/2}}{P_{bar}T_{m}\theta}$$
 Eq. 5-11

Where:

K' = Critical orifice coefficient, [m³)(°K)¹/₂]/